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MATERIALS FOR POTASSIUM LUBRICATED JOURNAL BEARINGS

Quarterly Progress Report No. 6
For Quarter Ending October 22, 1964

EDITED BY R. G. FRANK

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SPACE POWER AND PROPULSION SECTION

MISSILE AND SPACE DIVISION

GENERAL ELECTRIC

CINCINNATI, OHIO 45215

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QUARTERLY PROGRESS REPORT NO. 6

Covering the Period
July 22, 1964 to October 22, 1964

edited by

R. G. Frank
Program Manager

approved by

J. W. Semmel, Jr.
Manager, Materials and Processes

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

Contract NAS 3-2534

Technical Management
NASA - Lewis Research Center
R.L. Davies

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC COMPANY
CINCINNATI, OHIO, 45215

FOREWORD

The work described herein is being performed by the General Electric Company under the sponsorship of the National Aeronautics and Space Administration under Contract NAS 3-2534. Its purpose, as outlined in the contract, is to evaluate materials suitable for potassium lubricated journal bearing and shaft combinations for use in space system turbogenerators and, ultimately, to recommend those materials most appropriate for such employment.

R. G. Frank, Manager, Physical Metallurgy, Materials and Processes, is administering the program for the General Electric Company. L. B. Engel, Jr., D. N. Miketta, T. F. Lyon, W. H. Hendrixson and B. L. Moor are directing the program investigations. The design for the friction and wear testers was executed by H. H. Ernst and B. L. Moor.

R. L. Davies of the National Aeronautics and Space Administration is the technical manager for this study.

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I. INTRODUCTION

The program reviewed in this sixth quarterly report, covering activities from July 22, 1964 to October 22, 1964, is performed under the sponsorship of the National Aeronautics and Space Administration. Its purpose is to evaluate materials suitable for potassium lubricated journal bearing and shaft applications in space system turbogenerators operating over a 400^{o}F to 1600^{o}F temperature range. The critical role of bearings in such systems demands the maximum reliability attainable within today's state-of-the-art. Achieving this reliability requires an interdisciplinary approach employing the best mechanical designs of journal bearings combined with the selection of the optimum materials to serve as the structural members. Satisfying this latter requirement constitutes the aim of this program.

A number of investigators have conducted studies in this field and their contributions have advanced the state-of-the-art considerably (Section VIII, Ref. 1). Although their work is significant, there are no common criteria for a comparison of the existing data. Therefore, establishing a unified approach to the development and evaluation of materials for potassium lubricated bearing application is deemed essential. The program involves a comprehensive investigation of material properties adjudged requisite to reliable journal bearing operation in the proposed environment. This includes: 1) corrosion testing of individual materials and potential bearing couples in potassium liquid and vapor, 2) determination of hot hardness, hot compressive strength, modulus of elasticity, thermal expansion and dimensional stability characteristics, 3) wetting tests by potassium and 4) friction and wear measurements of selected bearing couples in high vacuum and in liquid potassium.

In cooperation with the cognizant NASA Technical Manager, fourteen candidate materials were selected (Table I) from a compilation of existing data on available materials. The materials reviewed fall into four broad categories:

- Superalloys and refractory alloys with and without surface treatment.
- Commercial metal bonded carbides.
- Refractory compounds such as stable oxides, carbides, borides and nitrides.
- Cermets based on the refractory metals and stable carbides.

Each material is procured from appropriate suppliers to mutually acceptable specifications and subsequently is subjected to chemical, physical and metallurgical analyses to document its characteristics before utilization in the program. After the documentation of processes and properties, the candidate materials undergo corrosion, dimensional stability, thermal expansion, compression and hot hardness testing. Considering the bearing material requirements and the preliminary information obtained on materials subjected to both potassium and non-potassium testing, a number of materials combinations will be selected in cooperation with and subject to the approval of the NASA Technical Manager. Potassium corrosion and wetting tests and friction and wear measurements in high vacuum and liquid potassium will then proceed with these combinations.

The ultimate product of this program will be a recommendation, substantiated with complete documentation, of the material or materials which have the greatest potential for use in alkali metal journal bearings in high speed, high temperature, rotating machinery for space applications. Hopefully, the results will indicate the future course of alloy or material development specifically designed for alkali metal lubricated journal bearing and shaft combinations.

TABLE I: CANDIDATE BEARING MATERIALS

Material Class	Candidate Material
A. Nonrefractory Metals and Alloys	Star J (17%W-32%Cr-2.5%Ni-3%Fe-2.5%C-Bal. Co)
B. Refractory Metals and Alloys	Mo-TZM (0.5%Ti-0.08%Zr-Bal. Mo) Tungsten
C. Fe-Ni-Co Bonded Carbides	Carboloy 907 (74%WC-20%TaC-6%Co) Carboloy 999 (97%WC-3%Co) K601 (84.5%W-10%Ta-5.5%C)
D. Carbides	TiC (94%)
E. Oxides	Lucalox (99.8% Al_2O_3) Zircoa 1027 (95.5% ZrO_2)
F. Borides	TiB_2 (98%)
G. Refractory Metal Bonded Carbides	TiC+5%W TiC+10%Mo TiC+10%Cb Grade 7178

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II. SUMMARY

During the sixth quarter of this program, the topics abstracted below were covered and the results are interpretatively presented in this report.

Essentially all the test specimens have been received and the over-all procurement status is 93% complete. The status of individual test specimen configurations is: corrosion, 100% complete; dimensional stability, 100% complete; thermal expansion, 97% complete; hot hardness, 100% complete; compression, 73% complete.

Approximately 25 pounds of potassium were purified by vacuum distillation at 500°F to 550°F and a receiver pressure of 2 x 10^{-5} torr. After additional purification by hot trapping in a titanium lined, zirconium gettered hot trap, the potassium will be used for the initial checkout tests of the friction and wear test rig facility.

Twenty Cb-1Zr alloy capsule assemblies were filled with purified potassium and tested isothermally for 1,000 hours at 800°F, 1200°F, and 1600°F in a vacuum of 10^{-8} to 10^{-9} torr. Chemical analyses, by the mercury amalgamation method, of samples of the potassium taken at the same time that the capsules were filled, indicated the potassium contained less than 50 ppm oxygen. Each of the three sets of six capsules contained test specimens of K601, TiC, 10%Cb+TiC, 5%W+TiC, Grade 7178, and Star J materials. Two additional capsules, tested at 1600°F, contained specimens of 10%Mo+TiC and TiB_2, respectively. The chamber pressure at the end of the test was 1 x 10^{-9} torr.

To evaluate dimensional stability, duplicate specimens of ten of the fourteen candidate materials were tested for 1,000 hours at 1200°F and 1600°F in vacuum. The chamber pressure at the conclusion of the test was 1.3 x 10^{-9} torr. Of the ten materials evaluated, the Zircoa 1027 was the only material to show a significant change in dimensions, i.e., approximately +0.4%, as a result of the 1,000-hour exposure at 1600°F. Slight dimensional changes, i.e., approximately 0.02%, were observed in Carboloy 999, Carboloy 907, and tungsten after exposure to both the 1200°F and 1600°F test temperatures and in K601 after exposure to the 1600°F test temperature. No detectable changes were observed for Lucalox, TiC, Mo-TZM, 10%Cb+TiC or Grade 7178. A second 1,000-hour test incorporating duplicate specimens of thirteen candidate materials has completed 750 hours at an 800°F test temperature and a chamber pressure of 1.1 x 10^{-9} torr.

Environmental check tests with Cb-1Zr alloy test specimens were concluded satisfactorily in the Chevenard dilatometer and the instrument was calibrated against a Pyros 56 standard. Subsequently, the the mean coefficient of thermal expansion was determined as a function

of temperature.

Author

of temperature for the following seven candidate materials: Mo-TZM, tungsten, Carboloy 999, Carboloy 907, Lucalox, Zircoa 1027, and Grade 7178. Again, dimensional instability was observed in the Zircoa 1027 material.

The construction of both the vacuum and liquid potassium friction and wear test rigs is proceeding satisfactorily and a considerable number of the components have been completed. During the next quarter, both test rigs are scheduled to be received, assembled, and installed and checkout tests are to be initiated with the vacuum test rig.

It was decided to proceed with the immersion conductive heater design for the liquid potassium friction and wear test rig. The heater incorporates a Cb-1Zr alloy sheath, Nichrome heating elements, and either BN or Al_2O_3 insulation. Although the high thermal conductivity and preliminary compatibility tests of BN and Cb-1Zr alloy at 1800°F indicate that BN should be an acceptable insulator in the immersion heater for the liquid potassium friction tester, a heater utilizing Al_2O_3 as the insulation also will be fabricated.

The detailed design of the wetting test facility was completed.

III. MATERIALS PROCUREMENT

Various test specimens were received throughout the quarter and at the end of the report interim the over-all procurement status was 93% complete (Table II). The following tabulation indicates the status of the individual specimen configurations:

- Corrosion - 100% Complete
- Dimensional Stability - 100% Complete
- Thermal Expansion - 97% Complete
- Hot Hardness - 100% Complete
- Compression - 73% Complete

Because of the complexity of the compression specimens, several vendors encountered considerable difficulty in producing the required number of specimens. At the end of the quarter, complete sets of ten compression specimens were on hand for eight of the fourteen candidate materials; partial deliveries were effected for three of the remaining six materials. Those specimens still to be delivered to achieve a 100% completion are listed below.

Material	Specimen Configuration	No. Ordered	No. Required to Complete Order
Star J	Thermal Expansion	3	2
Star J	Compression	10	10
K601	Compression	10	10
TiC	Compression	10	3
TiC+10%Mo	Compression	10	2
TiB_2	Compression	10	2
Tungsten	Compression	10	10

The vendor producing the 1.56-inch diameter, unalloyed tungsten bar stock, from which compression specimens will be machined, agreed to attempt to produce the material by direct extrusion from the 3.8-inch diameter billet to an approximate 1.75-inch diameter rod. The required 1.56-inch diameter size will be obtained by machining. This process will result in a 4.7:1 extrusion ratio or an approximate 79% reduction in area. The former method included a final rolling operation which gave an additional 25% reduction to the material, or a total reduction of 82%. On two previous attempts to roll to the 1.56-inch diameter rod, however, severe cracking was encountered. Because of the late delivery of the starting electrode stock and the scheduling difficulties which the subsequent conversion operations entailed, delivery of the material from the revised process was not firmly affixed at the end of the quarter.

Processing problems also were encountered in the production of dimensional stability specimens from Star J material. Repeated rejections of entire lots of specimens because of cracks detected by penetrant inspection precluded attempts to produce the specimens by sand casting techniques. This cracking problem was associated with the relatively large grain size and orientation of the carbide network, which resulted from the slow cooling achieved in the sand mold. Subsequent sectioning of the excess material from the casting and grinding to the finished product dimensions resulted in the severe cracking problem. It was found that a semi-chill casting technique (graphite mold) produced a grain structure more amenable to subsequent machining and grinding operations and specimens were produced by this casting technique. Any alteration in grain size and carbide morphology will be documented and factored into the final evaluation of the material.

Additional Mo-TZM alloy compression test specimens were ordered. These specimens, delivered in September, will be used to check out the compression test facility and to establish testing techniques. One of the Mo-TZM alloy compression test specimens was fabricated into an ultrasonic inspection standard to be used for inspecting all the compression specimens before mechanical testing. Two notches, each 0.500-inch long by 0.003-inch wide by 0.0038-inch deep (approximately 3% of reduced section wall thickness), were positioned longitudinally and circumferentially 90° apart in the center of the outside diameter of the reduced section. Inspection will employ circumferential and longitudinal shear wave techniques. In addition, a similar notch was placed circumferentially in the center of the radius of the reduced section to facilitate a careful survey of this critical area of the test specimen configuration.

TABLE II: PROCUREMENT STATUS OF THE CANDIDATE BEARING MATERIALS TEST SPECIMENS - (10-22-64)

			TEST SPECIMEN IDENTITY															
			CORROSION			DIMENSIONAL STABILITY			THERMAL EXPANSION			HOT HARDNESS			COMPRESSION			
Material	Spec.	Vendor	P.O. No.	Date Rec'd	Quantity	P.O. No.	Date Rec'd	Quantity	P.O. No.	Rec'd Or Promised	Quantity	P.O. No.	Date Rec'd	Quantity	P.O. No.	Rec'd Or Promised	Quantity	MCN Series
Carboloy 999	SPPS-24T	GE-MPD	037-121815	4-29-64	17	037-121815	4-29-64	10	037-122247	6-10-64	3	037-122247	6-10-64	3	037-122247	8-17-64	10	1035
Carboloy 907	SPPS-23T	GE-MPD	037-121815	4-29-64	18	037-121815	4-29-64	11	037-122247	6-10-64	3	037-122247	6-10-64	3	037-122247	8-17-64	10	1036
Mo-TZM (Raw Stock)	SPPS-15	Amer. Metal Climax Company	037-121885	3-6-64	0.437"øx48"	037-121885	3-6-64	1.25"øx36"	037-122249	5-5-64	0.187"øx12"	037-122249	5-5-64	0.750"øx12"	037-122249	5-5-64	1.56"øx36"	-
Mo-TZM (Machining)	-	Dawson Carbide Ind., Inc.	037-124116	4-29-64	16	037-124116	4-29-64	10	037-122284	6-30-64	3	037-122284	6-30-64	3	037-122284	7-6-64	10	1037
Tungsten (Raw Stock)	SPPS-40T	Univ. Cyclops Steel Corporation	037-122059	4-1-64	0.437"øx48"	037-122059	4-1-64	1.25"øx36"	037-122249	6-10-64	0.187"øx12"	037-122249	6-5-64	0.750"øx12"	037-122249	<u>11-23-64</u>	1.56"øx36"	-
Tungsten (Machining)	-	Dawson Carbide Ind., Inc.	037-124116	4-29-64	16	037-124116	4-29-64	10	037-122284	6-30-64	3	037-122284	6-30-64	3	2 Weeks after Receipt of Material			1038
Lucalox	SPPS-29T	GE-Lamp Glass	037-121835	5-4-64	16	037-121835	5-4-64	12	037-122243	7-7-64 7-12-64	2 1	037-122243	7-27-64	3	037-122243	8-21-64	10	1039
Zircoa 1027	SPPS-31T	Zirc. Corp of America	037-121868	5-4-64 6-12-64	11 5	037-121868	5-4-64	10	037-122248	6-12-64	3	037-122248	6-12-64	3	037-122248	10-12-64	10	1040
K601	SPPS-25T	Kennametal, Inc.	037-122216	6-10-64	17	037-122216	6-10-64 7-22-64	8 2	037-122245	7-22-64 8-21-64	2 1	037-122245	6-10-64	3	037-122245	<u>Indefinite Delivery</u>	10	1041
TiC	SPPS-27T	Kennametal, Inc.	037-121842	6-10-64 8-19-64	14 2	037-121842	6-10-64 7-22-64	9 1	037-122244	8-21-64	3	037-122244	7-22-64	3	037-122244	10-19-64 <u>Indefinite Del</u>	7 3	1042
TiC + 5%W	SPPS-33T	Kennametal, Inc.	037-121842	7-22-64	16	037-121842	7-22-64	10	037-122244	8-21-64	3	037-122244	8-21-64	3	037-122244	9-29-64	10	1043
TiC + 10%Mo	SPPS-34T	Kennametal, Inc.	037-121842	7-22-64	16	037-121842	7-22-64 8-21-64	8 2	037-122244	8-21-64	3	037-122244	7-22-64	3	037-122244	9-29-64	8	1044
TiC + 10%Cb	SPPS-35T	Kennametal, Inc.	037-121842	6-10-64 7-22-64	6 10	037-121842	6-10-64 8-21-64	4 6	037-122244	8-21-64	3	037-122244	8-21-64	3	037-122244	10-19-64	10	1045
Grade 7178	SPPS-36T	Kennametal, Inc.	037-122257	6-10-64 7-22-64	12 4	037-122257	6-10-64 7-22-64	9 1	037-122257	6-10-64	3	037-122257	6-10-64	3	037-122257	9-11-64	10	1046
Star J	SPPS-18T	Stellite Div., UCC	037-121822	6-19-64	16	037-121822	10-19-64	10	037-122248	<u>10-26-64</u>	3	037-122248	7-16-64	3	037-122248	<u>10-26-64</u> <u>Indefinite Del</u>	6 4	1047
TiB_2	SPPS-37T	Norton Company	037-121867	8-11-64 8-21-64	6 10	037-121867	8-21-64	10	037-122251	8-21-64	3	037-122251	8-21-64	3	037-122251	9-21-64 <u>10-26-64</u>	8 1	1048

IV. TEST FACILITIES

Thermal Expansion

The alterations to the Chevenard dilatomer described previously in Quarterly Progress Report No. 5,[2] resulted in an acceptable environment for the thermal expansion test program. The environment was evaluated by exposing a chemically cleaned 0.187-inch diameter Cb-1Zr alloy rod (Heat No. 373-4, Kawecki Chemical Company) to the anticipated test cycle, i.e., room temperature to 1600°F to room temperature (approximately 8 hours exposure time) and, then, establishing the magnitude of the increase in the gaseous elements in the Cb-1Zr alloy specimen.

The tested specimen was sectioned into three 0.125-inch thick transverse samples from the front, middle, and back of the rod and these were analyzed for oxygen, nitrogen, and hydrogen by the vacuum fusion method. Simultaneously, samples of the untested Cb-1Zr alloy rod were submitted for analyses to provide the pre-test interstitial levels. Table III presents the data obtained from the chemical analyses. From these data, it is clearly evident that a satisfactory test environment has been achieved in the test facility.

Hot Hardness

After the modifications described in Quarterly Progress Report No. 5[2] were completed, the hot hardness tester was re-assembled and evacuated. The pressure appeared to stabilize at approximately 2×10^{-5} torr after several hours. This improvement in the vacuum capabilities of the test facility was certified by exposing a Cb-1Zr alloy test specimen (No. 4) to the intended test cycle to 1600°F. The resultant hardness data (Table IV), which showed no increase in the surface hardness of the Cb-1Zr alloy, indicated a satisfactory test environment. To verify the quality of the environment further, the specimen was sectioned for chemical analyses of the interstitial elements oxygen, nitrogen, and hydrogen. Two thin sections of varying thickness were taken from the exposed micropolished surface and a third sample was taken from the center of the cube approximately 0.200 inch beneath the surface. All burrs were removed by polishing on silicon carbide paper and the samples were degreased in petroleum ether just before test. The approximate dimensions of each sample are tabulated on page 14.

TABLE III: CHEMICAL ANALYSES[1] OF Cb-1Zr ALLOY SPECIMENS[2] USED IN THE FINAL CHEVENARD DILATOMETER CHECKOUT TESTS

Sample Identity	Element, ppm O	N	H
Untested Material[3]	155	10	4
	171	9	5
Tested Material[4]			
Front Sample[5]	154	21	6
Middle Sample[5]	111	13	6
Back Sample[5]	133	14	6

[1] By vacuum fusion techniques.

[2] Specimen: MCN 1225-2, 0.187-inch diameter rod.

[3] Chemically cleaned (60% H_2O, 20% HNO_3, 20% HF) before analysis.

[4] Chemically cleaned before testing in dilatometer.

[5] Transverse section 0.125-inch thick.

TABLE IV: HOT HARDNESS DATA FOR Cb-1Zr ALLOY TEST SPECIMEN NO. 4
(Specimen: MCN 122, 0.5-inch Cube; One Surface Polished to Approximately 2-5 rms)

Time Minutes	Vacuum, torr	Temp., °F	Hardness Number, kg/mm²
--	--	RT	91[1]
--	--	RT	91[2]
1	1.8×10^{-5}	120	96
8	1.6×10^{-5}	209	83
13	3.4×10^{-5}	306	79
21	7.0×10^{-5}	400	76
35	8.0×10^{-5}	500	65
49	5.6×10^{-5}	600	65
63	5.0×10^{-5}	700	60
75	5.0×10^{-5}	800	58
87	6.0×10^{-5}	925	55
97	5.6×10^{-5}	1003	57
115	6.0×10^{-5}	1175	61
130	5.6×10^{-5}	1300	71
150	5.0×10^{-5}	1400	67
159	5.8×10^{-5}	1500	65
170	5.5×10^{-5}	1600	65
177	2.8×10^{-5}	1400	--
180	2.0×10^{-5}	1200	67
195	1.6×10^{-5}	983	65
230	1.0×10^{-5}	703	61
265	8.4×10^{-6}	500	67
--	--	RT	91[3]

NOTES:

1) Room temperature hardness of the sample on a Tukon tester using a 100-gram load:

91 kg/mm²
91 kg/mm²
92 kg/mm²
Average - 91 kg/mm²

2) Room temperature hardness of the sample on the hot hardness tester using a Vickers pyramid diamond, a 100-gram load, and a 15-second hold:

84 kg/mm²
96 kg/mm²
93 kg/mm²
Average - 91 kg/mm²

3) Room temperature hardness of the sample, after test cycle, on the hot hardness tester using a Vickers pyramid diamond, a 100-gram load, and a 15-second hold:

95 kg/mm²
90 kg/mm²
89 kg/mm²
Average - 91 kg/mm²

Sample Identity	Sample Dimensions	Remarks
A	0.024-inch thick x 0.119-inch wide x 0.347-inch long	One surface exposed to test environment
B	0.054-inch thick x 0.141-inch wide x 0.347-inch long	One surface exposed to test environment
C	0.106-inch thick x 0.186-inch wide x 0.188-inch long	Sample surface approximately 0.200 inch from surface exposed to test environment

The results of the analyses of the above specimens are presented in Table V.

TABLE V: CHEMICAL ANALYSES[1] OF Cb-1Zr ALLOY HOT HARDNESS SPECIMEN NO. 4[2]

Sample	Element, ppm O	N	H
A	290	17	22
B	187	18	6
C	156	20	4

[1] By vacuum fusion techniques

[2] MCN 122

The data show a concentration of oxygen on the exposed micropolished surface, evidenced by the dilution of the oxygen in Sample B versus the oxygen concentration in Sample A. However, since the average room temperature microhardness of the specimen before and after the exposure to the hot hardness tester conditions was identical, i.e., 91 kg/mm, the high oxygen concentration is probably restricted to a very thin layer at the surface and has no measurable influence on the bulk hardness of the Cb-1Zr alloy. Also, the candidate bearing materials are much less sensitive to environmental contamination than the Cb-1Zr alloy. For these reasons, it is believed that the modifications to the hot hardness tester have effected an acceptable environment for the test program. Testing of the candidate materials will be initiated in the next quarter.

Friction and Wear

Friction and Wear in Liquid Potassium. The major vendors of the tester components were visited several times during the reporting period to review their progress. In general, fabrication of the component parts is proceeding satisfactorily and on schedule; the majority of the materials, commercial items, and tooling are on hand and machining has been completed on a number of components.

Several items that were fabricated from refractory alloys have been welded at General Electric and returned to the vendor for finish machining. Included is the potassium exit tube, which comprises Type 304L SS tube, sleeves, and transition piece; a 3/4-inch schedule 80 Cb-1Zr alloy pipe; and a Cb-1Zr alloy/Type 304L SS bimetallic joint. The joint configuration is shown in Figure 1 and was fabricated according to Specification SPPS-9A, "Vacuum Brazing Bimetallic Tube Joints". Also included is the Cb-1Zr Alloy sump cover and baffle which has been welded, returned to the vendor, and fully machined. Semifinished machined components of the Cb-1Zr alloy disc specimen holder and the molybdenum sleeve assembly are currently being brazed at General Electric.

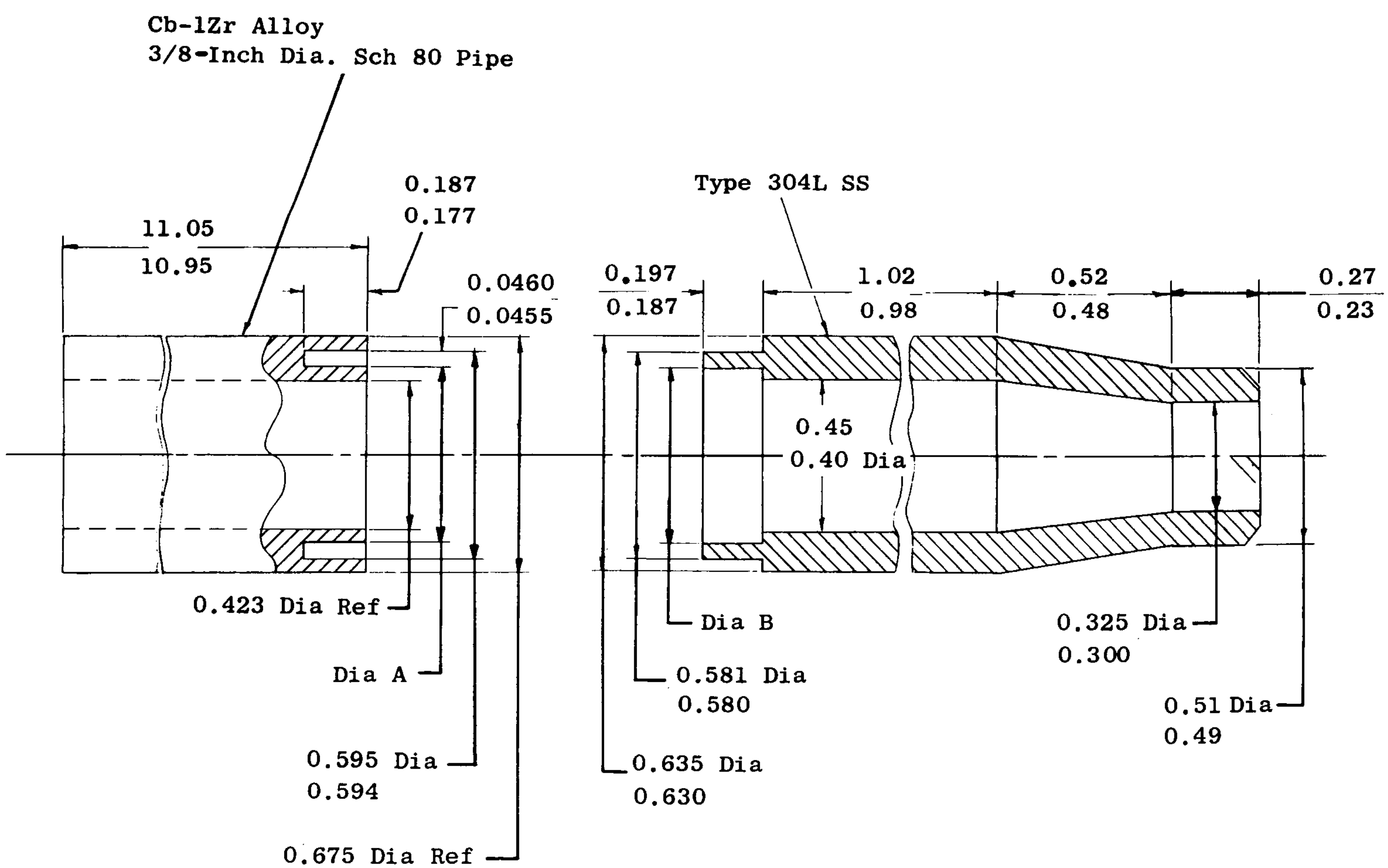
It was decided to proceed with the immersion, conductive potassium sump heater design consisting of a Cb-1Zr alloy sheath, Nichrome heating elements, and Al_2O_3 or BN insulation. Heaters with both types of insulation are being procured in the event that processing difficulties and/or interactions between the insulating material and the Cb-1Zr alloy sheath or Nichrome heating element prevent the fabrication or use of one. From the thermodynamic data shown in Table VI, both Al_2O_3 and BN are expected to be compatible with the sheathing and the heating element materials. Although the zirconium would be expected to react with BN to form ZrN, the small amount of free zirconium present in the Cb-1Zr alloy is not expected to affect the performance of the heaters. The alternate immersed radiation heater described in Quarterly Progress Report No. 5[2] could (not) be used without a major redesign of the test rig in order to satisfy the concurrent requirements of:

1) transmitting 500 amperes into the vacuum chamber,

2) allowing for the evacuation of the interior of the Cb-1Zr alloy heater assembly to approximately 10^{-7} torr,

3) preserving the capability of assembly and disassembly.

A supply of Cb-1Zr alloy tubing, required to make the sheaths for the fire rods, was located with a sufficiently short lead time that its delivery will not seriously delay the availability of the heater for assembly with the other components of the test rig. Standard components for making a stainless steel working model of the Cb-1Zr alloy sheathed heater will be ordered, however, so that assembly and vacuum and heat transfer checks may be made even though the delivery of the Cb-1Zr alloy heater is delayed.



1. Dia B to be .000 - .002 Loose in Dia A
2. Vacuum Braze to Specification SPPS-9A.

Figure 1. Bimetallic Joint Design for Cb-1Zr Alloy and Type 304 SS Pipe.

TABLE VI: HEATS, FREE ENERGIES AND ENTROPIES OF FORMATION[1]

Compound	$-\Delta H_{298}$	S_{298}	$-\Delta F_{300}$	$-\Delta F_{500}$	$-\Delta F_{1000}$	$-\Delta F_{1500}$	Accuracy[1] $\pm$ KCal
2/3 Al_2O_3	266.6	8.2	251.6	241.6	216.4	182.0	4
2/3 B_2O_3	203.9	8.6	191.4	182.9	---	---	4.5
2 CbO	195.0	---	---	176.6	---	---	4
CbO_2	190.9	13.03	177.5	168.5	146.2	123.8	2
2/5 Cb_2O_5	181.6	13.2	168.8	160.2	---	---	3
2 NiO	114.0	18.2	101.8	92.0	72.0	51.8	3
2/3 Cr_2O_3	180.0	12.9	167.4	157.8	137.2	116.4	4
ZrO_2	259.5	12.1	245.5	236.2	213.8	191.9	4
2 AlN	153.0	---	140.6	131.8	---	---	16
2 BN	128.4	7.34	188.2[2]	99.8[2]	78.6[2]	57.8[2]	10
2 CbN	113.6	---	100.0[3]	90.8[3]	---	---	4
2 Cb_2N	122.2	---	---	---	---	---	1
2 Ni_3N	- 0.4	---	---	---	---	---	1
2 CrN	58.8	---	46.2	37.6	19.4	---	3
2 Cr_2N	54.8	---	44.8	38.0	24.4	13.4	3
2 ZrN	174.6	18.6	160.6	141.8	129.4	106.6	3

(1) Smithells, Colin, J., "Metals Reference Book," Vol. II, Butterworth, Inc., Washington, D.C., 1962.

(2) JANAF Thermodynamic Tables, The Dow Chemical Company, Midland, Michigan, 1963.

(3) Elliot, J. F., "Thermochemistry for Steelmaking," Vol. I, Addison-Wesley Publishing Company, Inc., Massachusetts, 1960.

The following new designs or design changes to the test rig were made during the reporting period:

1) The flange with thermocouple well, shown in Figure 2, was designed to replace one of the loading arms so that the temperature of the potassium can be measured as it passes over the disc specimen surface.

2) The design of the power lead conduit for the immersion heating elements was changed to delete the solid insulation, which was to be packed and swaged inside the conduit tube, and to substitute a beaded insulation. This permits a dynamic vacuum to be achieved inside the heater during operation.

3) The fitting, shown in Figure 3, was designed to provide a welded primary vacuum seal in the test rig; insulation and quick disconnect of the electrical leads for the sump heater; and an opening for the evacuation of the heater conduit. Minor modifications of standard Conax fittings are required.

4) The electrical terminal, shown in Figure 4, was designed to prevent deformation of the power leads upon repeated assemblies.

5) The design of the shaft bearing, shown in Figure 5, was changed so that the direction of the thrust action of the upper bearing is reversed and, thus, convergent with the thrust direction of the lower bearing. Puller grooves also were designed in the shaft bearing assembly to facilitate disassemblies.

Other minor modifications were made so that mating hardware could accommodate the enumerated changes.

Cb-1Zr Alloy/BN Compatibility Test. One of the designs for the immersion conductive heater in the liquid potassium friction and wear test rig requires the use of BN as the insulator in the Cb-1Zr alloy sheathed, fire-rod heaters. Tests were initiated to evaluate the compatibility of the Cb-1Zr alloy in intimate contact with the BN at 1800^{o}F for time periods up to 100 hours.

The test fixture consisted of a 1/4-20 NC by 1-inch Cb-1Zr alloy bolt with a molybdenum nut on which were placed, successively, washers of 99.5% Al_2O_3, Cb-1Zr alloy, BN, Cb-1Zr alloy, and 99.5% Al_2O_3. The Cb-1Zr alloy specimens were 3/16-inch thick sections that were cut from 3/8-inch diameter schedule 80 pipe. Before testing, they were cleaned in a mixture of 60%H_2O-20%HNO_3-20%HF, rinsed in water followed

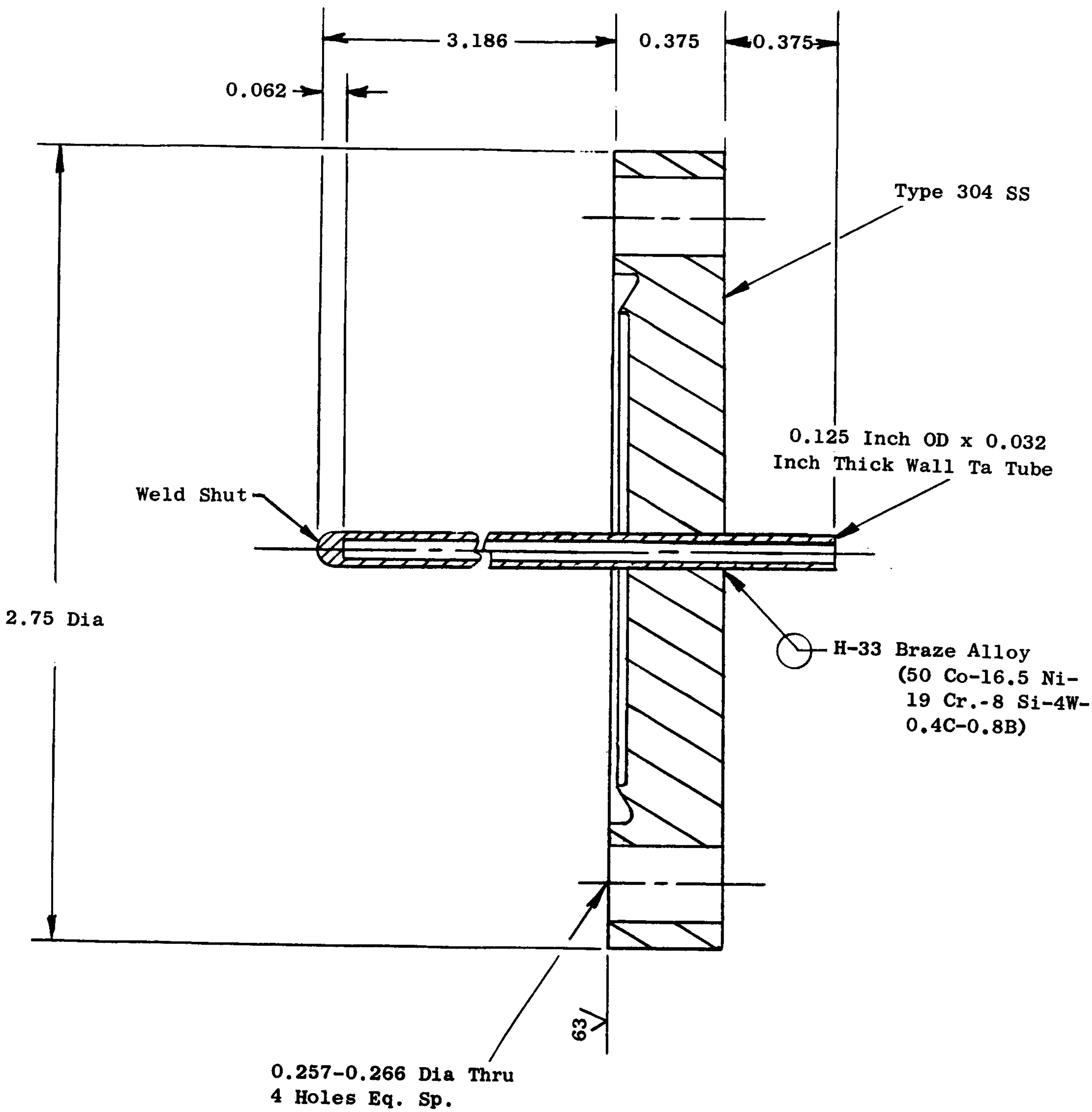


Figure 2. Flange Design with Thermocouple Well to be used to Measure Temperature of Potassium Flowing Over the Surface of the Disc Specimen in the Friction and Wear Tester.

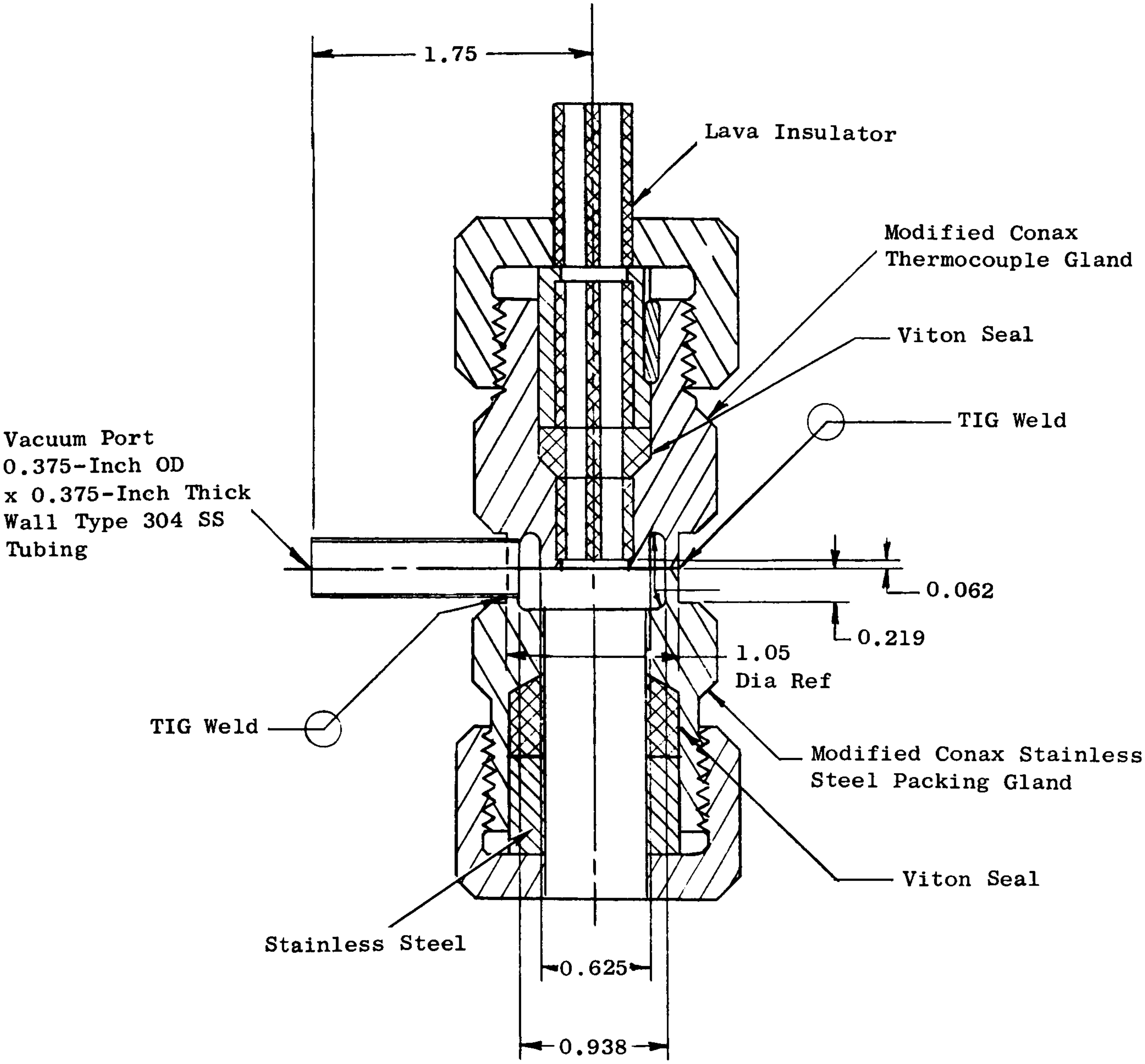


Figure 3. Power Feedthrough Fitting for Potassium Conductive Immersion Heater. The Primary Vacuum Seal to the System is Made by Means of a Conflat Flange Welded to the Conduit Tube.

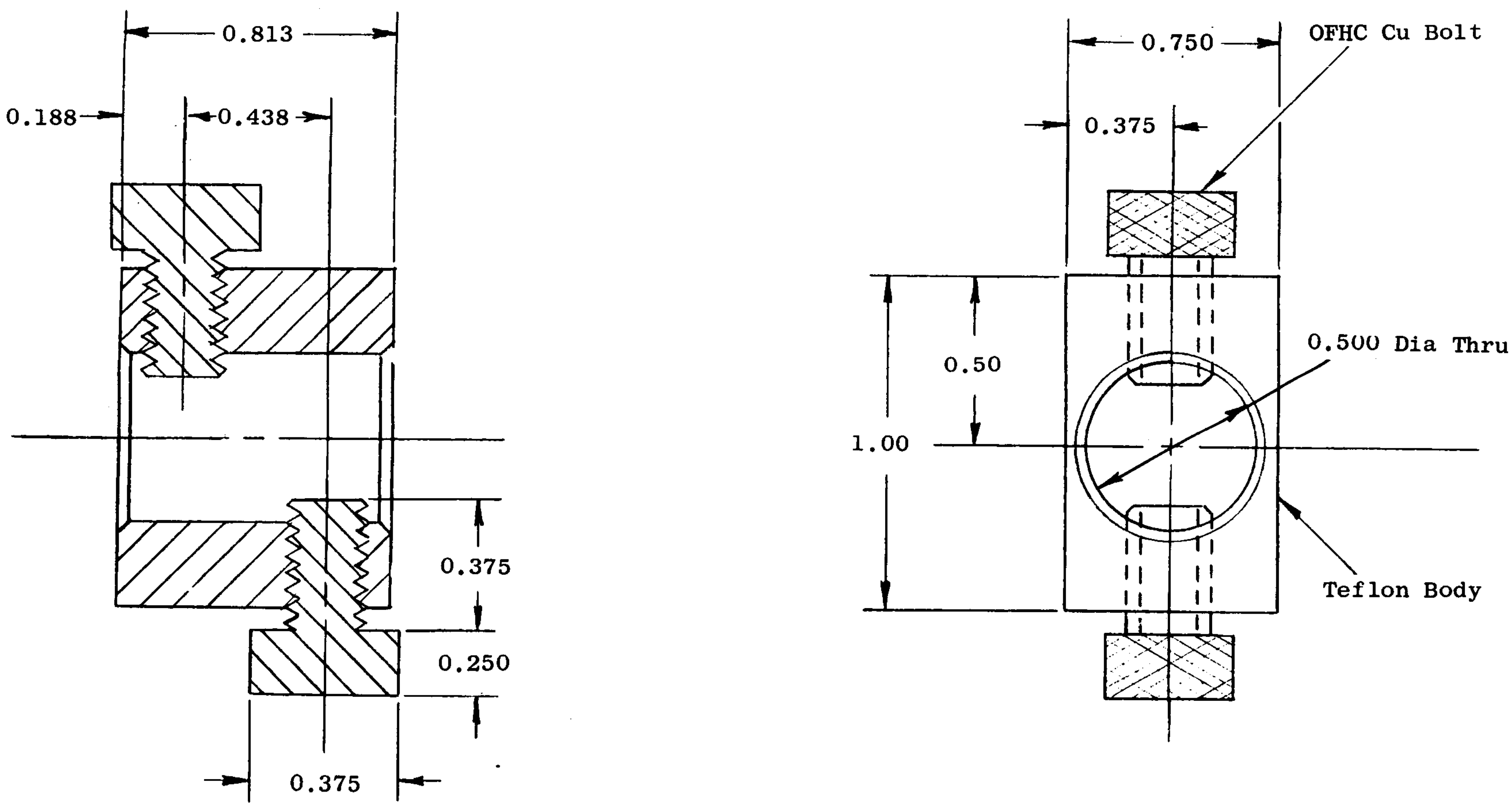
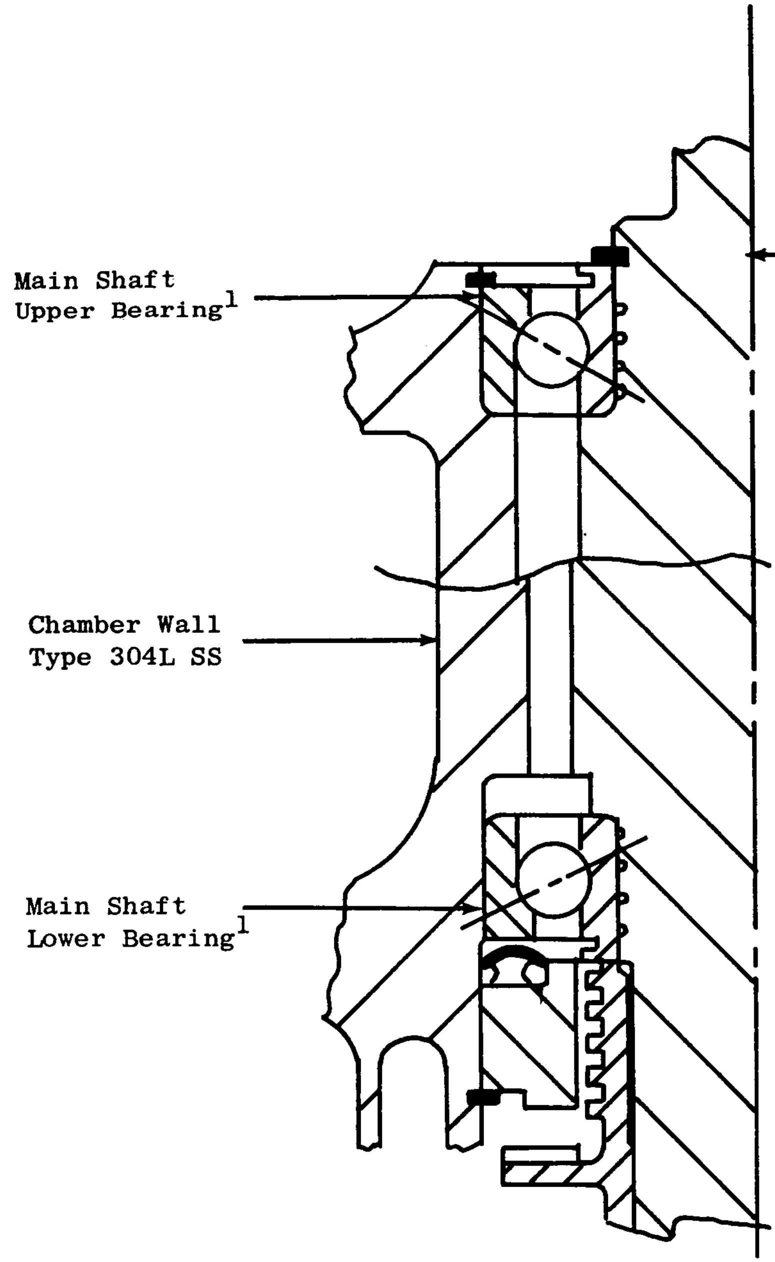


Figure 4. Electric Terminal for Power Leads to Potassium Conductive Immersion Heater.



[1]Vacuum Friction and Wear Test Rig
Balls - M50
Races - Au Plated M50
Separator - Ag Plated Circle C

Potassium Friction and Wear Test Rig
Balls - Type 440C SS
Races - Type 440C SS
Separator - Ni Plated Type 440C SS

Figure 5. Main Shaft Bearing Assembly.

by acetone, and air dried. The BN specimen was formed from a 1-inch diameter by 1/4-inch thick disc. Before testing, the BN specimen was given a vacuum bakeout for 19 hours at 675°F plus one hour at 2200°F at a pressure of 1×10^{-5} torr. The furnace used for the compatibility test consisted of a tantalum resistance element and was equipped with an oil diffusion pump with a water-cooled Chevron baffle between the pump and the test chamber. The furnace is capable of attaining a pressure of 5×10^{-6} torr.

After a Pt vs Pt+10%Rh thermocouple was attached to the Cb-1Zr alloy bolt head, the test fixture was wrapped in tantalum foil. The fixture was placed in the furnace on a tantalum support, and the system was sealed and evacuated to a pressure of less than 5×10^{-5} torr. The furnace was heated slowly to the test temperature of 1800°F, while the pressure in the furnace was maintained below 5×10^{-5} torr. After 15 hours at 1800°F, during which the pressure dropped to 5×10^{-6} torr, the furnace was turned off and allowed to cool to room temperature. The test fixture was removed from the furnace, a second assembly was installed, and the test was repeated under the same conditions for 100 hours.

The Cb-1Zr alloy test specimens were evaluated by chemical analyses (Table VII), metallography (Figure 6) and microhardness traverses (Figure 7). The increase in oxygen and the subsequent increase in surface hardness of the Cb-1Zr alloy are attributed to impurities in the BN that were not removed during the vacuum bakeout and, to a lesser extent, to environmental effects. Further evidence to this fact can be seen in the micrograph in Figure 6 showing the precipitation of ZrO_2 to the extent of approximately 0.012 inch from the surface. The pp't-free zone extending approximately 0.002 inch from the surface is attributed to the unavailability of free zirconium to react with the oxygen from the BN. Future tests are planned using BN, which has been baked out at 2800°F to remove the B_2O_3 impurities, or the higher purity BN that is now commercially available. Separate lots of the BN to be used in the fabrication of the initial "fire rods" will be outgassed at both 2200°F and 2800°F.

<u>Friction and Wear in High Vacuum.</u> Quotations were received on the high vacuum testers and the major components were ordered during the reporting period. The vendors include: McGregor Manufacturing Company (Troy, Michigan), the assembly and spare parts; Industrial Tectonics, Inc. (Compton, California), the bearings; Precision Mechanics, Inc. (Newton, Ohio), the loading arm assemblies. The scheduled delivery date for this machine is now December 15, 1964. The main shaft bearings will be the limiting item and an assembly made on that date will probably contain standard bearings. The over-all program is proceeding satisfactorily.

The shaft bearing design was changed in a manner similar to that noted for the potassium test rig. In addition, on the basis of experience at NASA-Goddard Space Flight Center,[3] decreasing the thickness of the gold plating in the races from 0.002-0.003 inch to 0.0003-0.0006 inch is being considered.

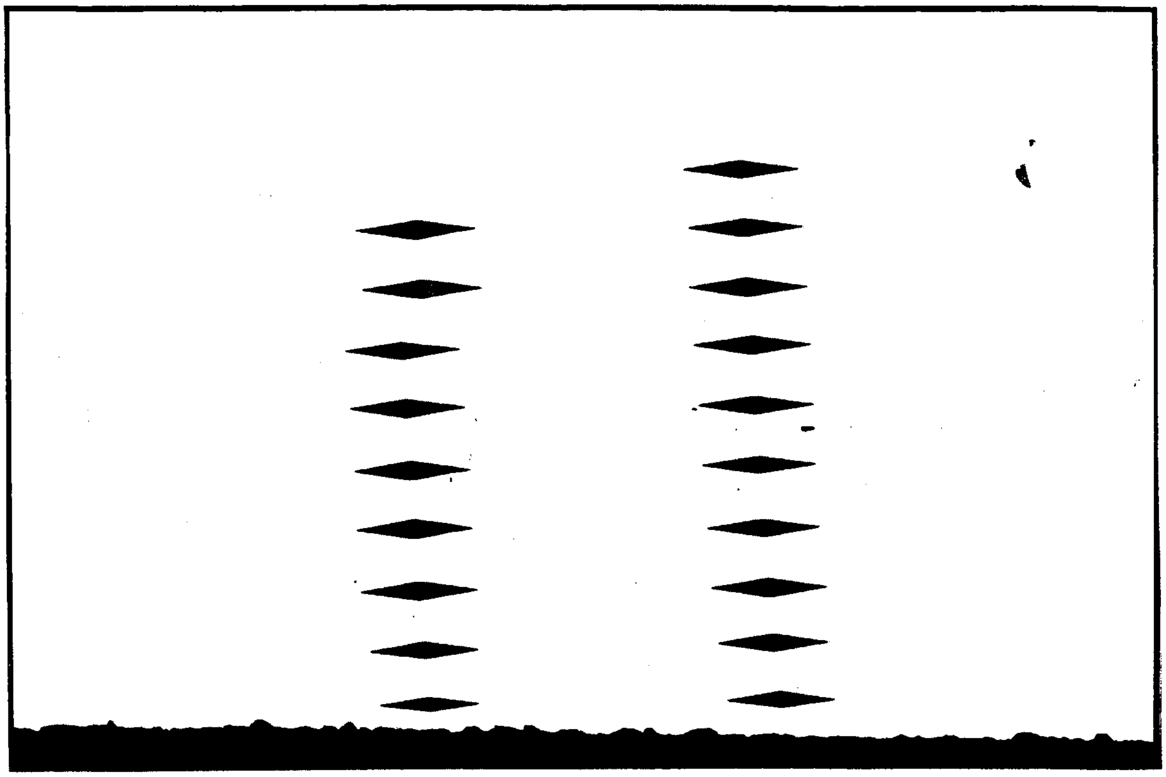
TABLE VII: CHEMICAL ANALYSES[1] OF Cb-1Zr ALLOY AFTER Cb-1Zr ALLOY/BN COMPATIBILITY TEST AT 1800°F IN VACUUM[2]

Sample Identity	Element, ppm		
	O	N	H
A. Cb-1Zr Alloy, As-Received	231	40	9
B. Cb-1Zr Alloy, After 15-Hour Exposure[3]	656	60	5
C. Cb-1Zr Alloy, After 100-Hour Exposure[3]	586	48	2

[1] By vacuum fusion techniques.

[2] 5×10^{-6} torr.

[3] Opposite surface exposed to Al_2O_3 was removed; specimen thickness 0.125 inch.



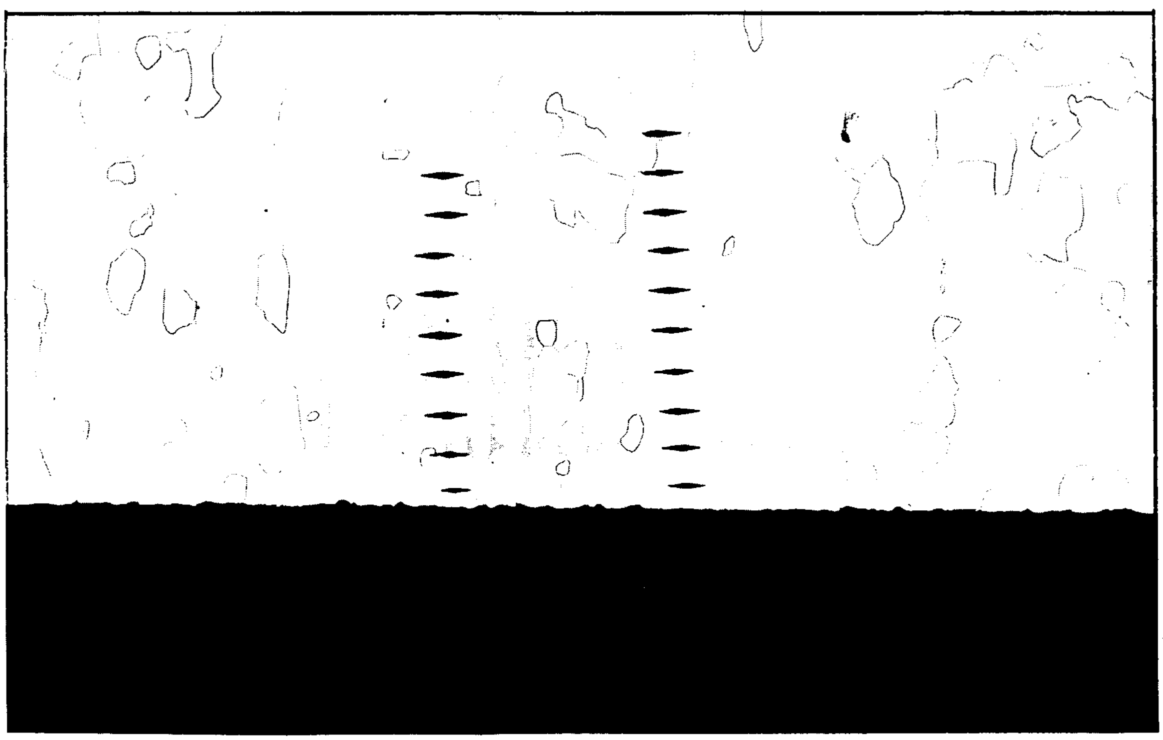


Figure 6. Microstructure of Cb-1Zr Alloy Surface in Contact with BN for 100 Hours at 1800°F in a Vacuum of 5×10^{-6} Torr.
Etchant: Top, As Polished — Mag: Top, 250X
Bottom, 20% HF, 20% HNO_3, 60% H_2O — Bottom, 100X

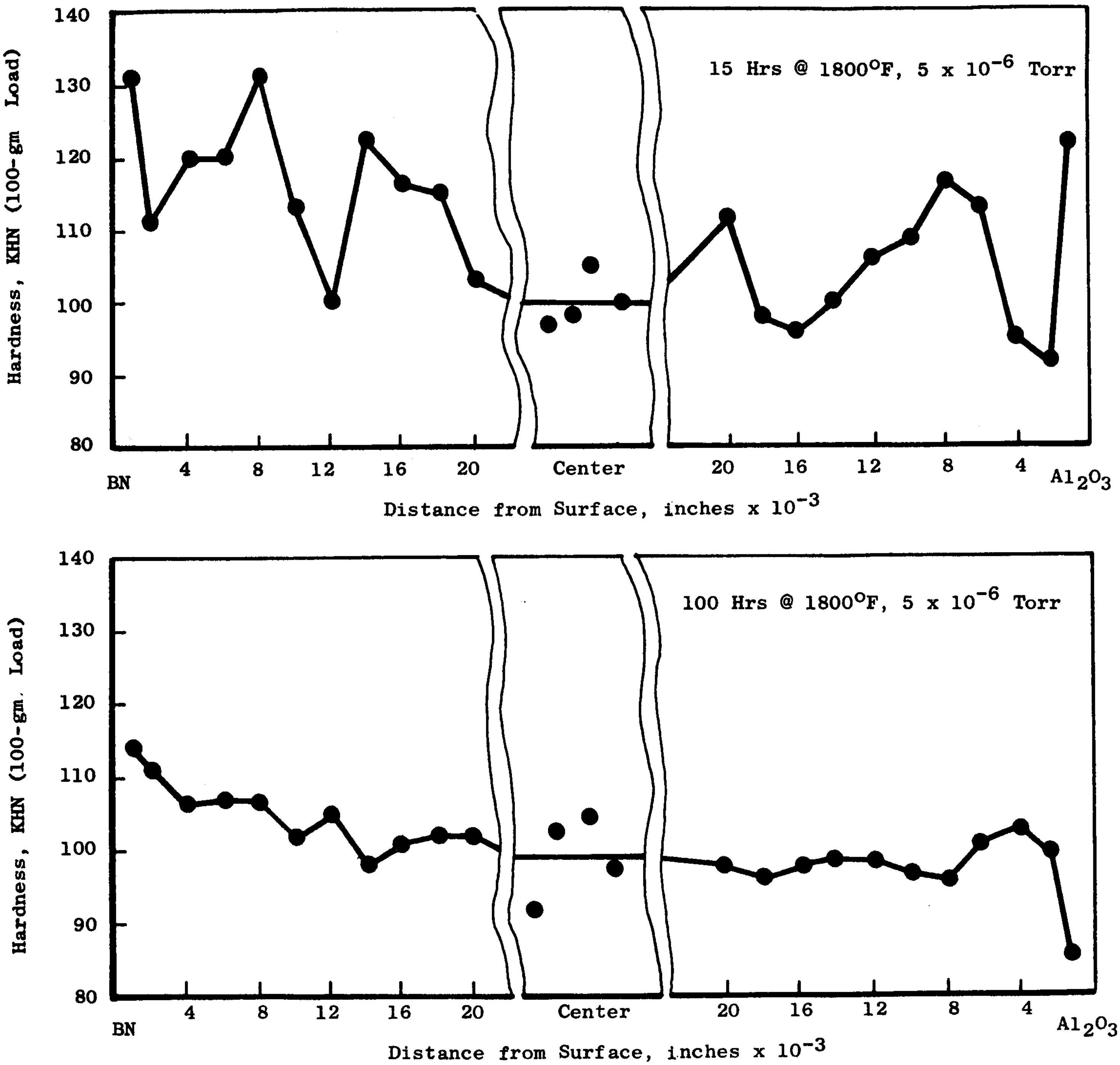


Figure 7. Microhardness Traverses of Cb-1Zr Alloy Specimens After Cb-1Zr Alloy/BN Compatibility Tests at 1800°F in Vacuum.

Materials Bonding Tests. During bakeout, operation, and, in the case of the potassium friction tester, potassium distillation, several materials combinations will be in intimate contact in an environment in which temperatures range up to 1600°F and pressures range as low as 10^{-9} torr. Two types of tests were initiated to evaluate the degree of bonding or seizing that would occur between the materials. Should such bonding occur, it would impede the operation of the friction tester or prevent the disassembly required for changing the specimens.

Screw Bonding Test. A screw bonding test was conducted to determine whether bonding would occur between the specimen holder and the shaft material or bolting material. If permitted to occur, such bonding would present a problem in changing specimens. The specimens used in the test, shown in Figure 8 and listed in Table VIII (Specimens No. 1 and No. 2), were 1/4-20 NC x 3/4-inch screws contained in drilled and tapped blocks. The equipment used to conduct the test consisted of a split element tantalum resistance heater with tantalum radiation shielding, which was installed in a Varian Model VI-16 high vacuum system as described in Quarterly Progress Report No. 3[4].

The specimens were appropriately cleaned, assembled, and tightened to 5 ft-lbs torque. The release torque was checked several times and was a consistent 4 ft-lbs in both material combinations. After final assembly, a Pt vs Pt+10%Rh thermocouple was attached to the specimens and the specimens were suspended in the center of the tantalum heater. Subsequently, the system was sealed, evacuated to a pressure of 4×10^{-6} torr, and given an 8-hour bakeout. After the bakeout, the pressure in the system, as measured by a Bayard-Alpert ionization gauge mounted on the chamber, was 2×10^{-9} torr.

The specimens were slowly heated to the 1600°F test temperature, while the pressure in the chamber was maintained below 1×10^{-7} torr. After reaching the test temperature, the specimens were held at 1600°F and an average chamber pressure of 5×10^{-8} torr for 2 hours and were then allowed to cool to room temperature. When the system was opened, the specimens were removed and the release torque was checked for evidence of any bonding of the screw. The Mo-TZM alloy screw was released from the Cb-1Zr alloy block at 5 ft-lbs torque, which was slightly higher than the 4 ft-lbs established before the test. The M-252 alloy screw in the Mo-TZM alloy block had loosened during the test and would have measured at something less than finger-tight. Visual examination produced no indication of specimen bonding. Although this situation might be different in the presence of potassium where surface oxides are reduced, previous tests[5] with vapor blasted Rene' 41 screws in a Type 316SS block, which were torqued to 11 ft-lbs and exposed to potassium for 500 hours at 1500°F in an Inconel capsule, also showed no evidence of bonding.

Bearing and Compression Bonding Tests. Addition materials combinations were investigated to determine whether bonding would occur because of a high vacuum bakeout or during the actual testing cycle. The specimens that were used in these tests are listed in Table VIII and are described on page 30.

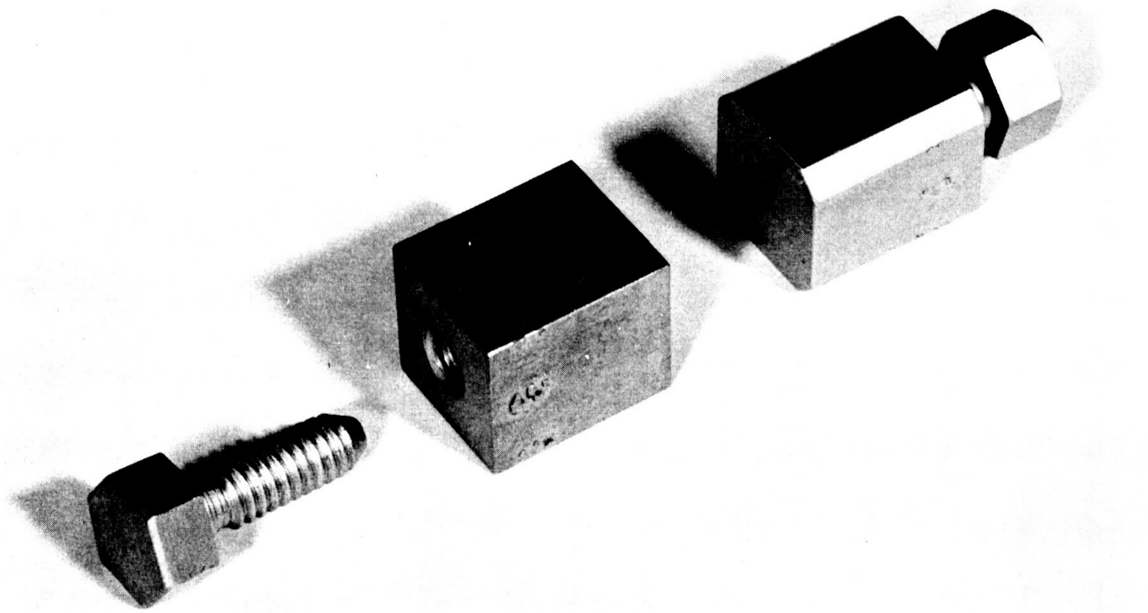


Figure 8. Screw Bonding Test Assemblies Tested in a Vacuum of 1 x 10^{-7} Torr for Two Hours at 1600°F: (Left) Mo-TZM Alloy Screw in Cb-1Zr Alloy Block and (Right) M-252 Alloy Screw in Mo-TZM Alloy Block. (C64090159)

TABLE VIII: MATERIALS BONDING TESTS

Test Number	Materials	Friction Tester Parts	Type of Test	Test Temp., °F	Time Hours	Vacuum Torr	Remarks Remarks
1	M-252 (Screw) vs Mo-TZM (Block)	Shaft, Specimen Holder Assembly	Screw	1600	2	5×10^{-8}	Pre-Test Torque 4 Ft-Lbs. Post-Test Torque 0 Ft-Lbs. No Evidence of Bonding.
2	Mo-TZM (Screw) vs Cb-1Zr (Block)	Specimen Holder, Screws	Screw	1600	2	5×10^{-8}	Pre-Test Torque 4 Ft-Lbs. Post-Test Torque 5 Ft-Lbs. No Evidence of Bonding.
3	M-50 (Ball) vs M-50 (Race)	Bearing Race, Balls	Bearing	750	16	2×10^{-7}	No Evidence of Bonding.
4	Type 304SS vs M-252	Shaft, Shims, Shields, Bearing Housing Bolts, Washers	Compression	750	16	2×10^{-7}	No Evidence of Bonding.
5	Type 304SS vs OFHC Cu	Flange, Gaskets	Compression	750	16	2×10^{-7}	Slight Bonding Occurred
6	Type 304SS vs SAE 1020 Steel	Bearing Retainer Shims, Bearing Housing	Compression	750	16	2×10^{-7}	No Evidence of Bonding
7	Type 304SS vs Type 304SS	Bearing Housing, Shims	Contact	750	16	2×10^{-7}	No Evidence of Bonding.

Specimen No.	Description
3	Two ball bearing assemblies of similar materials but of different size. Each bearing was mounted in a compression fixture that produced, by bolt tension, a simulated axial load of approximately 60 lbs on the bearing.
4	Discs of each material clamped together in a compression fixture.
5 and 6	Heavy washers of each of the test materials bolted together on a stainless steel bolt.
7	1-inch by 1-inch by 3/8-inch thick pieces of the materials loosely stacked together without external pressure.

The equipment and test setup were the same as those described for the screw bonding test except that a larger heater and radiation shielding was required to accommodate the increased number of specimens. Although a preliminary test was conducted to determine whether the chamber bakeout system could be used as the heat source for the test, the maximum internal chamber temperature attained was only 350°F and that approach was discarded.

The test assemblies were installed in the chamber, and the system was sealed and evacuated to a pressure of 4×10^{-6} torr. After an 8-hour bakeout period, the chamber pressure was 2×10^{-8} torr. Then, the specimens were slowly heated to the test temperature of 750°F, measured by a Pt vs Pt+10%Rh thermocouple attached to one of the bearing specimens. Although an attempt was made to maintain the chamber pressure in the 10^{-8} torr range during the test, due to outgassing of the system, the lowest pressure attained during the 33-hour heat-up period was in the 10^{-7} torr range with short bursts as high as 1×10^{-5} torr. The specimens were held at the test temperature of 750°F for 16 hours at an average pressure of 2×10^{-7} torr.

After cooling to room temperature, the test setup was disassembled and each specimen was examined for evidence of bonding. The only specimen that had bonded was the OFHC copper washer vs the Type 304 SS washer, which represented a flange and sealing gasket. However, examination of the mating surfaces revealed no evidence of any welding that would have scarred the surface of a flange. In application, the copper gasket will be replaced before each assembly. All other assemblies showed no evidence of bonding.

Wetting

Detailed design of the potassium wetting apparatus has been completed. Engineering drawings of the facility are currently being prepared and will be forwarded to the NASA Technical Manager for approval during the next report interim.

V. TEST PROGRAM

Potassium Purification

An additional 24.9 pounds of potassium from container D-80 was transferred to the modified container attached to the still and was outgassed at 400°F to 450°F for one hour before distillation. No gas evolution caused by the large conductance of the vacuum manifold was detected. The entire 24.9 pounds of potassium was distilled at 500°F to 550°F with the receiver pressure at approximately 2×10^{-5} torr; the initial pressure in the receiver, before heating the still for distillation, was 1×10^{-6} torr. As before, the distillate was bright and looked very clean.

After further purification by hot trapping, the purified potassium will be used to fill capsules for the third series of 1,000-hour corrosion tests and for the initial checkout of the potassium friction and wear test rig.

Corrosion

Twenty previously prepared Cb-1Zr alloy capsule assemblies were filled with purified potassium, sealed under vacuum using the same procedures described in Quarterly Progress Report No. 5,[2] and tested for 1,000 hours at 800°F, 1200°F, and 1600°F in a vacuum of 10^{-8} to 10^{-9} torr. Each of three sets of six capsules contained test specimens of the following six materials:

- K601
- Grade 7178
- TiC
- TiC+5%W
- TiC+10%Cb
- Star J

Two additional capsules, containing specimens of TiC+10%Mo and TiB_2, respectively, were tested at 1600°F.

Before filling the capsules, samples of the potassium were taken directly from the hot trap in lengths of stainless steel pipe and analyzed for oxygen content by the mercury amalgamation method. The level of oxygen as K_2O was 5 ppm. After the first set of capsules were filled, a faulty valve on the hot trap had to be replaced. A second sample then taken for analysis contained 12.3 ppm oxygen as K_2O. In both cases, the oxygen value represents the average of duplicate analyses from which no blank has been subtracted. Samples of potassium were also obtained during the filling of the four sets of corrosion capsules and were analyzed for oxygen by the mercury amalgamation method. Table IX gives these results. Radiographic examination of the capsules after filling with potassium indicated that all twenty capsules had been filled to the proper level. However, two of the capsules lacked full penetration welds on the top end caps and had to be repair welded.

A recent investigation[6] of the amount of oxygen contamination caused by grit blasting with Al_2O_3, conducted to specification SPPS-12, "Grit Blasting Columbium and Columbium Alloy Products," has shown an increase of 842 ppm oxygen on 0.0175-inch thick Cb-1Zr alloy sheet. Based on these data, it was decided not to grit blast the twenty capsules before testing.

The assembled corrosion capsules were installed in the corrosion test facility located in the Varian high vacuum chamber, C-IV. The capsules were instrumented with Pt vs Pt+10%Rh thermocouples on both the top and bottom end caps. Two thermocouples, which had been recalibrated at zinc, aluminum, and copper melting points, were installed on capsules in each susceptor and will be recalibrated after 1,000 hours of testing.

The chamber was sealed, evacuated to 3.8×10^{-7} torr, and given a 24-hour bakeout at 400°F after which the pressure dropped to 1.5×10^{-9} torr. All four susceptors were brought to their respective test temperature simultaneously, while the chamber pressure was maintained at less than 1×10^{-6} torr. Nineteen hours after the test temperatures were achieved, the pressure in the chamber was 7.2×10^{-8} torr. Figure 9 plots the pressure change during test. All pressure values are obtained from a tubular Bayard-Alpert ionization gauge located on the side of the chamber. The mean test temperatures and their deviations are given in Table X.

The vacuum distillation apparatus for cleaning the corrosion test specimens, described in Quarterly Progress Report No. 5,[2] is 90% complete. Checkout of the facility will be completed during the next report interim.

Dimensional Stability

Test Run No. 1. The test setup and testing procedures used for the first dimensional stability test are described in Quarterly Progress Report No. 5.[2] The test was conducted for 1,000 hours without interruption with the vacuum well below the required 10^{-7} torr range and the test temperatures within $\pm$ 1%.

During the initial heat-up of the susceptors, the pressure, measured with a Tubular Bayard-Alpert ionization gauge, reached a maximum of 8×10^{-7} torr. The pressure dropped to the 10^{-8} torr range very rapidly and, at the conclusion of the test, was 1.3×10^{-9} torr. The change in pressure during the test is plotted in Figure 10.

TABLE IX: CHEMICAL ANALYSES[1] OF POTASSIUM USED FOR ISOTHERMAL CAPSULE CORROSION TESTS

Sample Identity	Sample Location	Sample Weight, gm		Oxygen[2] Content, ppm
Capsule Nos.	Cast Inside	1.155[3]		10.7
29, 30, 32,	EB Tank	1.654[3]		2.4
35, 38, 41		2.014[3]		3.2
			Avg.	4.75[4]
Capsule Nos.	From Transfer	2.184[3]		9.0
29, 30, 32,	Line	1.037[3]		6.5
35, 38, 41				
			Avg.	4.84[4]
Capsule Nos.	Cast Inside	2.240[3]		14.6
31, 33, 36,	EB Tank	3.585[3]		6.6
39, 42, 43				
			Avg.	9.68[4]
Capsule Nos.	Cast Inside	3.840[5]		37.4
34, 37, 40	EB Tank	2.146[5]		21.6
			Avg.	31.7[4]
Capsules No.	Cast Inside	4.188[5]		16.3
44, 45, 46,	EB Tank	2.139[5]		12.7
47, 48				
			Avg.	15.1[4]

[1] By mercury amalgamation method.

[2] As K_2O.

[3] Argon cover gas used during analyses.

[4] Average calculated by total μgrams oxygen/total sample weight with no blank being subtracted.

[5] Helium cover gas used during analyses.

TABLE X: TEST TEMPERATURES FOR SECOND 1,000-HOUR ISOTHERMAL CAPSULE CORROSION TEST

Suspector Number	Capsule Numbers	Test Temp., ^oF	Mean Temp., ^oF	Mean Temp. Deviation ^oF	Mean Temp. Deviation %
1	29, 32, 35, 38, 41, 44	800	803	$\pm$ 19.3	$\pm$ 2.41
2	30, 33, 36, 39, 42, 45	1200	1202	$\pm$ 10.7	$\pm$ 0.88
3	31, 34, 39, 40, 43, 46	1600	1597	$\pm$ 8.1	$\pm$ 0.51
4	47, 48	1600	1599	$\pm$ 20.0	$\pm$ 1.24

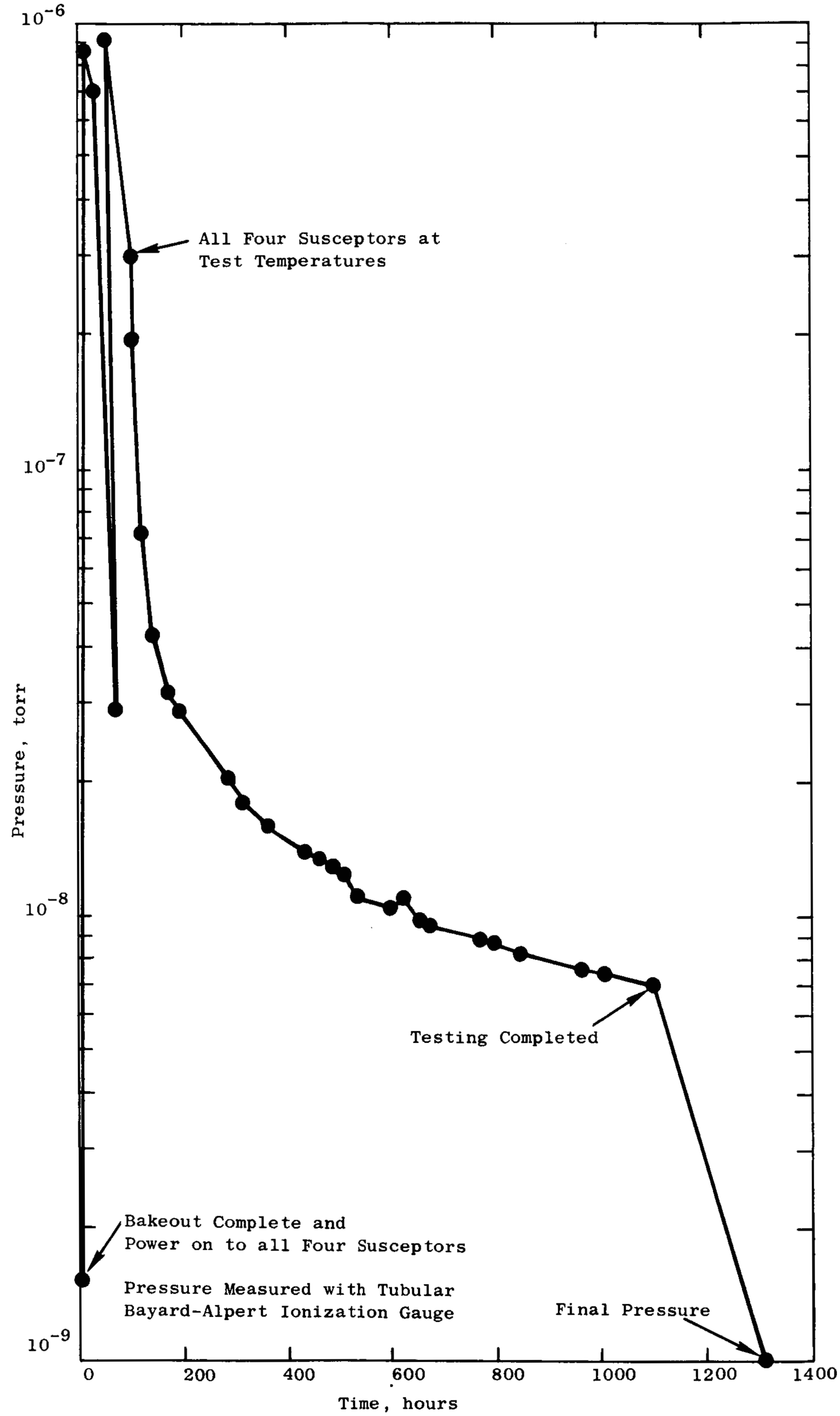


Figure 9. Pressure Curve for Second 1,000-Hour Isothermal Capsule Corrosion Test.

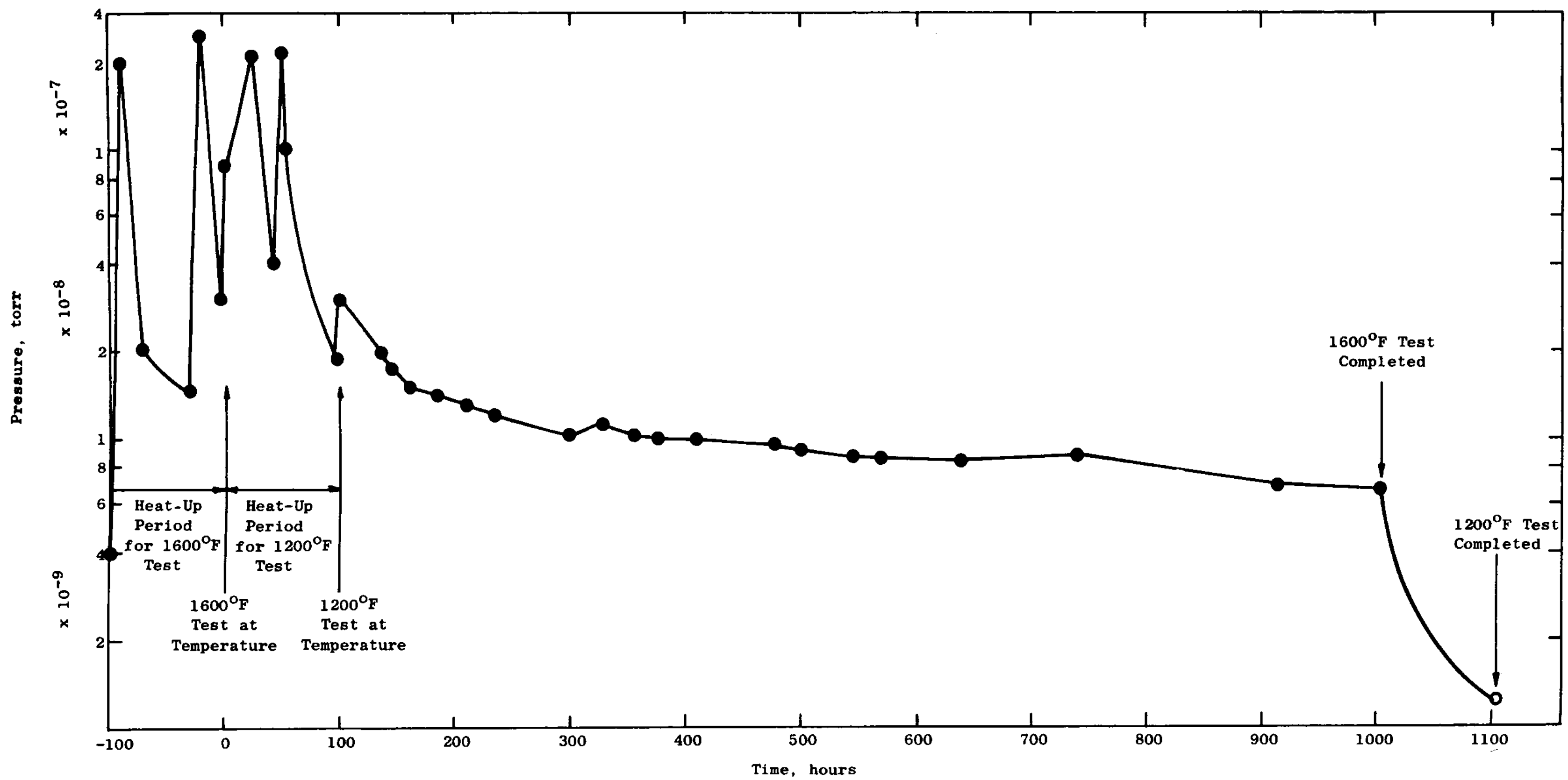


Figure 10. Pressure Curve for Dimensional Stability Test No. 1.

Average temperature readings and mean temperature deviations obtained during the test with a Leeds and Northrup potentiometer are shown in Figures 11 and 12. The instrumentation and thermocouple locations are described in the Quarterly Progress Report No. 5.[2] Figure 11 shows that, after the test temperature was stabilized, a a reasonably constant temperature was maintained throughout the 1200°F test. The lower average temperature in the last 100 hours, i.e., 1171°F, coincided with the termination of the 1600°F test. Some difficulty, attributed to the coarse manual control on the power supply, was experienced in maintaining the second susceptor at the 1600°F test temperature. Vibration over a period of time probably caused the temperature to drift. However, the test temperature still was maintained within $\pm$ 1%.

Two Pt vs Pt+10%Rh thermocouples, one installed in each susceptor, were checked against the National Bureau of Standards' freezing points in a standard calibration furnace before and after the test. These checks indicated no significant change in the temperature readings of the thermocouples after the 1,000-hour test, i.e., no measurable change for the thermocouple exposed for 1,000 hours at 1200°F and less than a + 0.3°F change for the thermocouple exposed for 1,000 hours at 1600°F.

Three pieces of 0.060-inch thick Cb-1Zr alloy sheet were placed on the top of three of the top specimen boxes in each susceptor before the susceptor covers were installed. To determine the amount of environmental contamination, selected samples from each susceptor were analyzed for oxygen, nitrogen, hydrogen, and carbon. These data are presented in Table XI.

Specimen Evaluation Test Run No. 1. All the specimens that were exposed for 1,000 hours at 1200°F and 1600°F have been weighed and measured dimensionally. To check the precision of the measurements, an untested Carboloy Grade 999 specimen (MCN-1035-B-9) was measured with test specimens both before and after the test. The largest deviation between measurements of the untested specimen was 0.04 x 10^{-3} inch. Therefore, a change in dimension no greater than 0.04 x 10^{-3} inch in any test specimen was considered no measurable change. Tables XII and XIII give the differences in the dimensional measurements of the test specimens before and after test.

Based on these data, the Zircoa 1027 (ZrO_2) was the only material of the ten evaluated to show a significant dimensional instability, i.e., approximately + 0.4%, and this occurred at the 1600°F test temperature. Slight dimensional changes, i.e., approximately 0.010 to 0.02%, were observed after the 1,000-hour exposures for Carboloy 999, Carboloy 907, and tungsten at both the 1200°F and 1600°F test temperatures and for K601 at the 1600°F test temperature. The remaining five materials appear to be quite stable, particularly Lucalox (Al_2O_3). Because several measurements were believed to be

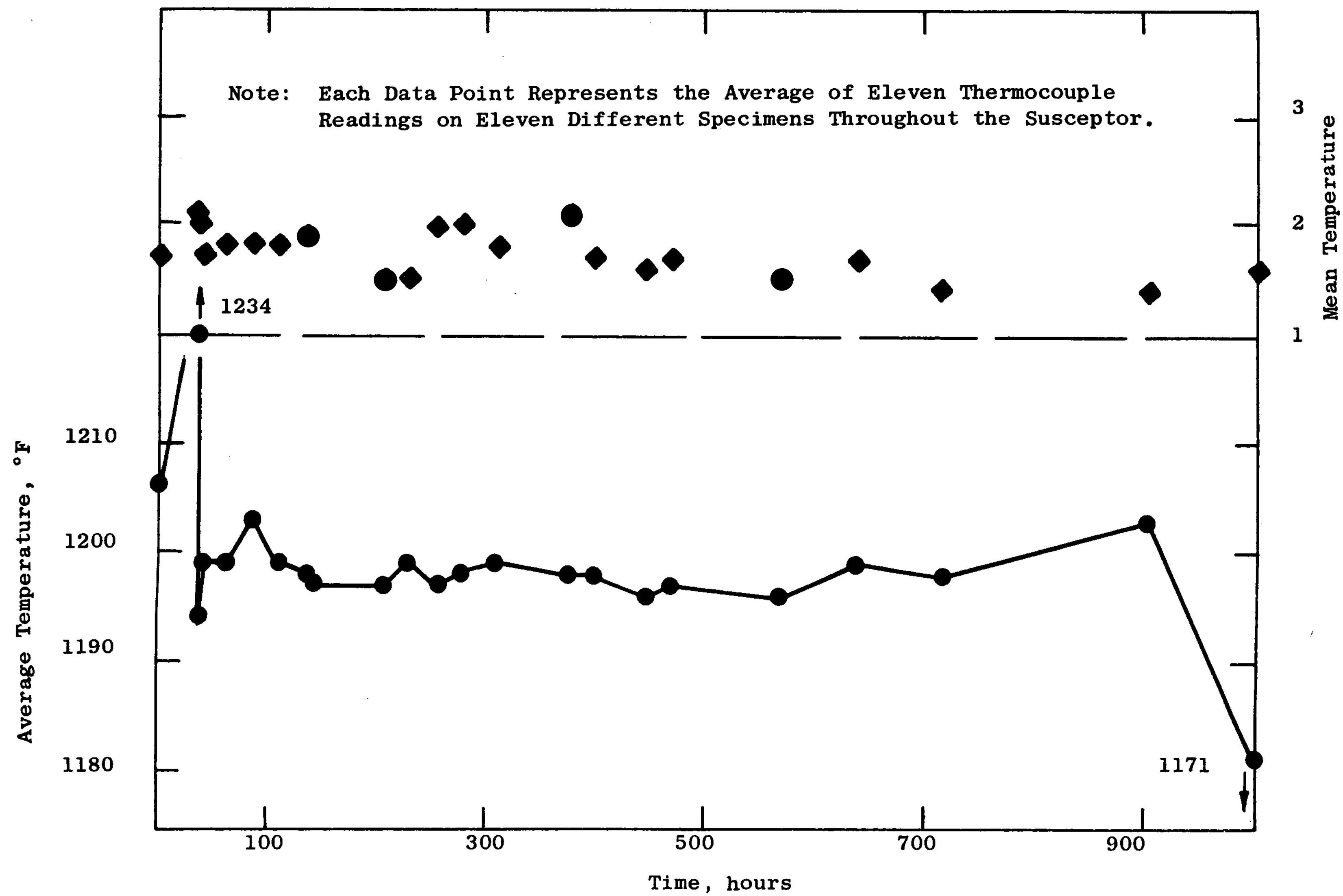


Figure 11. Average Temperature for Dimensional Stability Test No. 1, 1200°F.

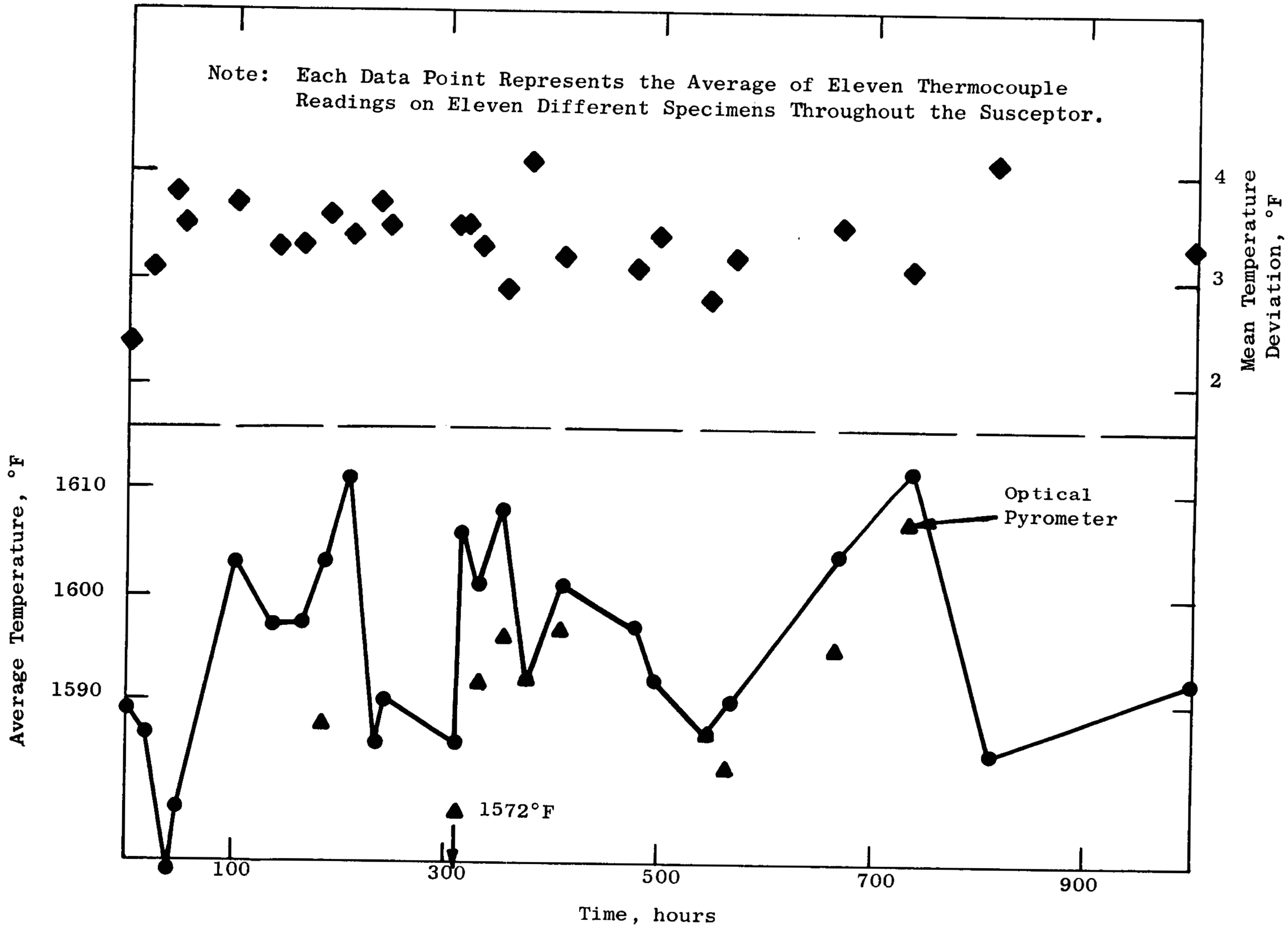


Figure 12. Average Temperature for Dimensional Stability Test No. 1, 1600°F.

TABLE XI: CHEMICAL ANALYSES OF Cb-1Zr ALLOY SHEET[1] EXPOSED IN VACUUM[2] FOR 1,000 HOURS AT 1200° AND 1600°F DURING DIMENSIONAL STABILITY TEST NO. 1

Sample Identity	Element, % O	N	H	C
Sample Exposed in 1600°F Susceptor	0.0115	0.0049	0.0003	0.002
	- - -	- - -	- - -	0.003
Sample Exposed in 1200°F Susceptor	0.0095	0.0046	<0.0001	0.001
	- - -	- - -	- - -	0.001
Before Test	0.0084	0.0040	0.0001	0.003
	0.0075	0.0040	0.0001	0.003

[1]MCN 418-2, 0.060-inch thick sheet.

[2]Ref. Figure 10.

TABLE XII: DIMENSIONAL AND WEIGHT CHANGES OF SPECIMENS EXPOSED IN VACUUM FOR 1,000 HOURS AT 1200°F

(Dimensional Stability Test Run No. 1)

Specimen Identity		Specimen Length, Inches		Dimensional Change	Dimensional Change	Specimen Width, Inches		Dimensional Change	Dimensional Change	Specimen Height, Inches		Dimensional Change	Dimensional Change	Specimen Weight in Grams		Weight Change
Material	MCN No.	Before Test	After Test	Inches x 10^{-3}	Percent	Before Test	After Test	Inches x 10^{-3}	Percent	Before Test	After Test	Inches x 10^{-3}	Percent	Before Test	After Test	Milligrams
Carboloy Grade 999	1035-B-3	1.00265	1.00224	-0.41	-0.041	0.89792	0.90790	+9.98	+1.100	0.79790	0.79790	0.00	0.000	178.9482	178.9487	+0.5
	1035-B-3[3]	1.00265	1.00275	+0.10	+0.010	0.89792	0.89797	+0.05	+0.005	0.79790	0.79795	+0.05	+0.006			
	1035-B-4	1.00237	1.00275	+0.38	+0.038	0.90225	0.89800	-4.25	-0.470	0.80223	0.79800	-4.23	-0.528	180.8079	180.8081	+0.2
	1035-B-4[3]	1.00237	1.00247	+0.10	+0.010	0.90225	0.90230	+0.05	+0.006	0.80223	0.80242	+0.19	+0.024			
Carboloy Grade 907	1036-B-3	1.00164	1.00184	+0.20	+0.020	0.90260	0.90276	+0.16	+0.018	0.80286	0.80302	+0.16	+0.020	174.9400	174.9399	-0.1
	1036-B-4	1.00116	1.00128	+0.12	+0.012	0.90089	0.90103	+0.14	+0.016	0.80274	0.80299	+0.15	+0.019	174.5221	174.5223	+0.2
Mo-TZM[1]	1037-B-3	1.00164	1.00166	+0.02	+0.002	0.90249	0.90249	0.00	0.000	0.80218	0.80218	0.00	0.000	120.6738	120.6731	-0.7
	1037-B-4	1.00315	1.00317	+0.02	+0.002	0.90239	0.90243	+0.04	+0.004	0.80248	0.80249	+0.01	+0.001	120.9059	120.9058	-0.1
Tungsten[2]	1038-B-3	1.00191	1.00195	+0.04	+0.004	0.90198	0.90190	+0.08	+0.009	0.80265	0.80250	-0.15	-0.019	228.7422	228.7423	+0.1
	1038-B-4	1.00216	1.00218	+0.02	+0.002	0.90245	0.90235	-0.10	-0.011	0.80269	0.80250	-0.19	-0.024	228.8816	228.8822	+0.6
Lucalox (Al_2O_3)	1039-B-3	1.00255	1.00255	0.00	0.000	0.90267	0.90266	-0.01	-0.001	0.80195	0.80194	-0.01	-0.001	47.1627	47.1627	0.0
	1039-B-4	1.00206	1.00205	-0.01	-0.001	0.90213	0.90211	-0.02	-0.002	0.80215	0.80213	-0.02	-0.002	47.0888	47.0886	-0.2
Zircoa 1027 (ZrO_2)	1040-B-3	1.00276	1.00278	+0.02	+0.002	0.90278	0.90283	+0.05	+0.006	0.80062	0.80034	-0.28	-0.035	67.8484	67.8480	-0.4
	1040-B-4	1.00160	1.00166	+0.06	+0.006	0.90184	0.90159	-0.25	-0.028	0.80132	0.80134	+0.02	+0.002	67.7924	67.7917	-0.7
K601	1041-B-3	1.00260	1.00268	+0.08	+0.008	0.90266	0.90271	+0.05	+0.006	0.80218	0.80225	+0.07	+0.009	187.7521	187.7523	+0.2
	1041-B-4	1.00278	1.00284	+0.06	+0.006	0.90243	0.90249	+0.06	+0.007	0.80214	0.80221	+0.07	+0.009	187.8038	187.8042	+0.4
TiC	1042-B-3	0.99927	0.99926	-0.01	-0.001	0.89734	0.89736	+0.02	+0.002	0.79817	0.79820	+0.03	+0.004	57.9604	57.9604	0.0
	1042-B-4	1.00275	1.00274	-0.01	-0.001	0.90207	0.90206	-0.01	-0.001	0.80182	0.80183	+0.01	+0.001	58.7377	58.7377	0.0
TiC + 10% Cb	1045-B-3	1.00245	1.00253	+0.08	+0.008	0.90234	0.90240	+0.06	+0.007	0.80260	0.80267	+0.07	+0.009	60.8978	60.8975	-0.3
	1045-B-4	1.00243	1.00251	+0.08	+0.008	0.90230	0.90237	+0.07	+0.008	0.80260	0.80267	+0.07	+0.009	60.9046	60.9046	0.0
Grade 7178	1046-B-3	1.00271	1.00276	+0.05	+0.005	0.90228	0.90236	+0.08	+0.009	0.80236	0.80241	+0.05	+0.006	172.9144	172.9144	0.0
	1046-B-4	1.00266	1.00271	+0.05	+0.005	0.90243	0.90249	-0.06	-0.007	0.80242	0.80247[4]	+0.05	+0.006	172.9294	172.9295	+0.1
	1046-B-4[3]	1.00266	1.00269	+0.03	+0.003	0.90243	0.90241	+0.02	+0.002	0.80242	0.80243	+0.01	+0.001			

1 Stress Relieved 3/4 Hour at 2350°F

2 Stress Relieved 1 Hour at 2000°F

3 Specimen Remeasured

4 This First Measurment was Reported (By Metrollogy Lab) as 0.82470, But After Remeasurement of the Specimen, it is Believed that the First Figure was in Error and Should have been 0.80247, as Indicated.

TABLE XIII: DIMENSIONAL AND WEIGHT CHANGES OF SPECIMENS EXPOSED IN VACUUM FOR 1,000 HOURS AT 1600°F

(Dimensional Stability Test Run No. 1)

Specimen Identity		Specimen Length, Inches		Dimensional Change	Dimensional Change	Specimen Width, Inches		Dimensional Change	Dimensional Change	Specimen Height, Inches		Dimensional Change	Dimensional Change	Specimen Weight in Grams		Weight Change
Material	MCN No.	Before Test	After Test	Inches x 10^{-3}	Percent	Before Test	After Test	Inches x 10^{-3}	Percent	Before Test	After Test	Inches x 10^{-3}	Percent	Before Test	After Test	Milligrams
Carboloy Grade 999	1035-B-1	1.00274	1.00287	+0.13	+0.013	0.90166	0.90176	+0.10	+0.011	0.80215	0.80221	+0.06	+0.007	180.7742	180.7734	-0.8
	1035-B-1[3]	1.00274	1.00287	+0.13	+0.013	0.90166	0.90164	-0.02	-0.002	0.80215	0.80205	-0.10	-0.012			
	1035-B-2	0.99717	0.99729	+0.12	+0.012	0.89782	0.90791	+10.09	+1.102	0.79720	0.79735	+0.15	+0.019	177.9098	177.9089	-0.9
	1035-B-2[3]	0.99717	1.00730	+0.13	+0.013	0.89782	0.89800	+0.18	+0.020	0.79720	0.79735	+0.15	+0.019			
	1035-B-11[4]	1.00215	1.00220	+0.05	+0.005	0.90229	0.90233	+0.04	+0.004	0.79824	0.79828	+0.04	+0.005	179.8239	179.8227	-1.2
Carboloy Grade 907	1036-B-1	1.00201	1.00208	+0.07	+0.007	0.90280	0.90285	+0.05	+0.005	0.80276	0.80279	+0.03	+0.004	174.9719	174.9697	-2.2
	1036-B-2	0.99875	0.99879	+0.04	+0.004	0.90194	0.90206	+0.12	+0.013	0.80259	0.80264	+0.05	+0.006	174.2532	174.2506	-2.6
Mo-TZM[1]	1037-B-1	1.00173	1.00179	+0.06	+0.006	0.90238	0.90241	+0.03	+0.003	0.80239	0.80243	+0.04	+0.005	120.7277	120.7274	-0.3
	1037-B-2	1.00247	1.00250	+0.03	+0.003	0.90261	0.90265	+0.04	+0.004	0.80174	0.80177	+0.03	+0.004	120.7320	120.7317	-0.3
	1037-B-9[4]	1.00238	1.00237	-0.01	-0.001	0.90271	0.90267	-0.04	-0.004	0.80257	0.80255	-0.02	-0.002	120.8468	120.8469	+0.1
Tungsten[2]	1038-B-1	1.00069	1.00074	+0.05	+0.005	0.90281	0.90282	+0.01	+0.001	0.80243	0.80245	+0.02	+0.002	228.6022	228.6022	0.0
	1038-B-2	1.00194	1.00200	+0.06	+0.006	0.90186	0.90185	-0.01	+0.001	0.80241	0.80254	+0.13	+0.016	228.6648	228.6650	+0.2
Lucalox (Al_2O_3)	1039-B-1	1.00236	1.00236	0.0	0.000	0.90250	0.90250	0.0	0.000	0.80266	0.80268	+0.02	+0.002	47.2000	47.2000	0.0
	1039-B-2	1.00186	1.00186	0.0	0.000	0.90186	0.90189	+0.03	+0.003	0.80219	0.80220	+0.01	+0.001	47.1496	47.1496	0.0
	1039-B-12[4]	1.00197	1.00195	-0.02	-0.002	0.90230	0.90230	0.0	0.000	0.80237	0.80230	-0.07	-0.009	47.2052	47.2054	+0.2
Zircoa 1027 (ZrO_2)	1040-B-1	1.00278	1.00661	+3.83	+0.383	0.90245	0.90614	+3.69	+0.409	0.80203	0.80523	+3.20	+0.399	68.1148	68.1147	-0.1
	1040-B-1[3]	1.00278	1.00668	+3.90	+0.390	0.90245	0.90621	+3.76	+0.417	0.80203	0.80529	+3.26	+0.407			
	1040-B-2	1.00265	1.00670	+4.05	+0.405	0.90215	0.90583	+3.68	+0.426	0.80142	0.80460	+3.18	+0.397	68.0733	68.0728	-0.5
	1040-B-2[3]	1.00265	1.00684	+4.19	+0.419	0.90215	0.90589	+3.84	+0.409	0.80142	0.80468	+3.26	+0.407			
K601	1041-B-1	1.00273	1.00284	+0.11	+0.011	0.90267	0.90271	+0.04	+0.004	0.80272	0.80280	+0.08	+0.099	188.0619	188.0618	-0.1
	1041-B-2	1.00255	1.00265	+0.10	+0.010	0.90257	0.90268	+0.11	+0.012	0.80217	0.80229	+0.12	+0.015	187.7841	187.7839	-0.2
TiC	1042-B-1	1.00273	1.00269	-0.04	-0.004	0.90205	0.90200	-0.05	-0.006	0.80247	0.80245	-0.02	-0.002	58.7738	58.7729	-1.1
	1042-B-2	1.00271	1.00271	-0.03	-0.003	0.90205	0.90200	-0.05	-0.006	0.80216	0.80214	-0.02	-0.002	58.7418	58.7410	-0.8
TiC+10%Cb	1045-B-1	1.00247	1.00250	+0.03	+0.003	0.90201	0.90203	+0.02	+0.002	0.80265	0.80268	+0.03	+0.004	60.8782	60.8772	-1.0
	1045-B-2	1.00244	1.00251	+0.07	+0.007	0.90236	0.90238	+0.02	+0.002	0.80261	0.80266	+0.05	+0.006	61.0743	61.0731	-1.2
Grade 7178	1046-B-1	1.00239	1.00243	+0.04	+0.004	0.90252	0.90255	+0.03	+0.003	0.80276	0.80279	+0.03	+0.004	170.1047	170.1047	0.0
	1046-B-1[3]	1.00239	1.00244	+0.05	+0.005	0.90252	0.90252	0.0	0.000	0.80276	0.80279	+0.03	+0.004			
	1046-B-2	1.00266	1.00270	+0.04	+0.004	0.90243	0.90245	+0.02	+0.002	0.80252	0.80251	-0.01	-0.001	172.9001	172.8997	-0.4
	1046-B-2[3]	1.00266	1.00273	+0.07	+0.007	0.90243	0.90245	+0.02	+0.002	0.80252	0.80250	-0.02	-0.002			

1 Stress Relieved 3/4 Hour at 2350°F
2 Stress Relieved 1 Hour at 2000°F
3 Specimen Remeasured
4 Stress-Relieved 1 Hour at 1800°F in Vacuum Prior to Test

erroneous, check measurements were made on the Carboloy 999 and Grade 7178 specimens and the results are considered valid. Check measurements also made on Zircoa 1027 specimens confirmed the original data.

The Zircoa 1027 was the only material to exhibit a visual change in appearance after testing. The as-received specimens had a light brown marbleized appearance; after exposure, the brown color changed to a clear light yellow over most of the specimen, with dark brown spots remaining at the point of contact between the specimens and the Cb-1Zr alloy wires of the Cb-1Zr alloy specimen boxes. This change in color was more pronounced in the specimens tested at 1600°F. If the brown color is assumed to be associated with the Fe_2O_3 impurity in the Zircoa 1027, the change in color could be due to the dissociation of Fe_2O_3 to Fe_3O_4 + oxygen at the low pressure of the test according to the following equation,[7]

$$\text{Log } P_{O_2} \text{ (mm)} = -\frac{A}{T} + B$$

where

P_{O_2} = partial pressure of oxygen

A = -24,912

B = +17.281

T = temperature, °K

As indicated by the negligible change observed in all these specimens, the 1800°F stress-relief treatment after the final grinding operation had no apparent influence on the dimensional changes recorded for the Mo-TZM alloy and Lucalox. However, the stress-relief may have some beneficial effects on the stability of the Carboloy 999 material.

Test Run No. 2. Duplicate specimens of thirteen of the fourteen candidate bearing materials were cleaned, measured, and weighed as described in previous progress reports and placed on test at 800°F. Although the test was delayed several weeks to include specimens of all fourteen materials, difficulties encountered in producing the Star J specimens made their delivery uncertain and it was necessary to start the test with the thirteen materials on hand.

The specimens were placed in the susceptor in the sequence shown in Table XIV. After the susceptor was loaded, the vacuum system was closed and evacuated to 2×10^{-6} torr. The system was then given a 10-hour bakeout at 550°F. After the bakeout cycle, the pressure was 3×10^{-8} torr, as measured by a Bayard-Alpert ionization gauge, and the susceptor temperature was 250°F. Subsequently, the susceptor

TABLE XIV: LOCATION OF SPECIMENS FOR DIMENSIONAL STABILITY TEST RUN NO. 2
(800°F TEST)

Specimen	Column 1	Column 2	Column 3	Column 4	Column 5
1 (Top)	TiC+5%W MCN 1043-B-2*	Grade 7178 MCN 1046-B-5*	Zircoa MCN 1040-B-5*	TiB_2 MCN 1048-B-6*	Tungsten MCN 1038-B-6*
2	Lucalox MCN 1039-B-6	TiC+10%Cb MCN 1045-B-5*	TiC+10%Mo MCN 1044-B-1*	TiC MCN 1042-B-6	Lucalox MCN 1039-B-5
3	TiB_2 MCN 1048-B-5	K601 MCN 1041-B-5	Carboloy 999 MCN 1035-B-5	Tungsten MCN 1038-B-5	K601 MCN 1041-B-6
4	Mo-TZM MCN 1037-B-5	TiC+10%Mo MCN 1044-B-2	Carboloy 999 MCN 1035-B-6	Grade 7178 MCN 1046-B-6	Mo-TZM MCN 1037-B-6
5	TiC MCN 1042-B-5	TiC+5%W MCN 1043-B-1*	Carboloy 907 MCN 1036-B-6*	Zircoa MCN 1040-B-6*	Carboloy 907 MCN 1036-B-6*
6 (Bottom)	TiC+10%Cb MCN 1045-B-6	Mo	Mo	Mo	Mo

*Indicates thermocouples are attached to these specimen boxes.

was slowly brought up to temperature while the pressure was maintained below 1 x 10^{-6} torr. Seventy-seven hours were required to reach 800°F. At the end of the reporting period, the test had accumulated 750 hours with the pressure at 1.1 x 10^{-9} torr. The average temperature of twelve thermocouples attached to the specimen boxes (Table XIV) was 796°F with a mean deviation of $\pm$ 4.4°F.

Thermal Expansion

Before initiating the test program, the dilatometer was calibrated by establishing the coefficients of thermal expansion for the Pyros 56 standard and comparing the data with the published values for that alloy (Table XV). Table XVI and Figure 13 present the thermal expansion data obtained for the Pyros 56 alloy in the initial run and in subsequent checks interposed during the testing of the candidate bearing materials to document the reproducibility and accuracy of the Chevenard instrument. The data evolved from the check tests of the Pyros 56 material indicate a most satisfactory performance of the equipment.

In setting up the tests, each specimen, the push rods, and the quartz specimen holders were cleaned in alcohol and thoroughly dried before their assembly; also, lint free gloves were worn when handling the specimen and equipment. The specimen and the Pyros 56 standard were inserted into their respective quartz tubes and the instrument was purged for one hour with the ultra pure helium. After purging for one hour, the exit stop-cock was closed and a positive pressure of helium was maintained within the system. Then, the dilatometer was zeroed by adjusting the linkage between the push rods and recording pen.

A heating and cooling rate of 570°F to 580°F per hour was used in all the tests. Temperatures were checked periodically with a calibrated Pt vs Pt+10%Rh thermocouple positioned on the exterior of the quartz tubes at the midpoint of the specimen and Pyros 56 standard. Temperature readout was accomplished using a calibrated potentiometer. In all tests to date, the variance between the thermocouple measurement and the temperature indicated by the actual thermal expansion curve, as plotted by the Pyros 56 standard, was within $\pm$ 10°F. The temperature was recorded continuously on a strip chart between the periodic check points.

The thermal expansion data for seven of the fourteen candidate materials are presented in Tables XVII through XXIII and Figures 14 through 22. The materials tested were Mo-TZM, tungsten, Carboloy 999, Carboloy 907, Lucalox, Zircoa 1027, and Grade 7178. Of those tested, only Zircoa 1027 showed evidence of instability. The remaining materials indicated a remarkable reproducibility in the heating and cooling curves of identical specimens tested on separate days. Note that test dates of duplicate runs for any one material were approximately one to two weeks apart. This procedure was selected to provide a secondary check test of the accuracy of the instrument.

Measurements of all the specimens were made before and after testing with a micrometer capable of reading to the fourth decimal place. Both specimens of the Zircoa 1027 material decreased 0.0005 inch in length. Testing of the seven remaining candidate material will be completed in the next quarter.

TABLE XV: MEAN COEFFICIENT OF THERMAL EXPANSION OF PYROS 56[1]

Test Temp., °F	Mean Coefficient of Thermal Expansion, in./in./°F x 10^{-6}
77-392	7.57
77-572	7.79
77-752	8.08
77-932	8.36
77-1112	8.62
77-1292	8.89
77-1472	9.12
77-1562	9.24
77-1652	9.36

[1]Pyros 56 data supplied with Chevenard dilatometer manual "Instructions for Assembly and Operation of Chevenard Mechanical Dilatometer," R.Y. Ferner Co., Inc., Malden, Mass.

TABLE XVI: CALIBRATION TESTS OF THE CHEVENARD DILATOMETER USING A PYROS 56 STANDARD

Test No. 1
Date: 8-18-64

Test Temp., °F	Mean Coefficient of Thermal Expansion, in./in./°F x 10^{-6}		
	Heating	Cooling	Average
77-392	7.50	7.50	7.50
77-512	7.74	7.74	7.74
77-752	8.08	8.08	8.08
77-932	8.35	8.35	8.35
77-1112	8.67	8.67	8.67
77-1292	8.90	8.90	8.90
77-1472	9.10	9.10	9.10
77-1562	9.21	9.21	9.21
77-1607	9.33	9.33	9.33

Test No. 2
Date: 9-3-64

Test Temp., °F	Mean Coefficient of Thermal Expansion, in./in./°F x 10^{-6}		
	Heating	Cooling	Average
77-392	7.75	7.75	7.75
77-512	7.95	7.95	7.95
77-752	8.08	8.08	8.08
77-932	8.41	8.41	8.41
77-1112	8.67	8.67	8.67
77-1292	8.88	8.88	8.88
77-1472	9.10	9.10	9.10
77-1562	9.25	9.25	9.25
77-1607	9.30	9.30	9.30

Test No. 3
Date: 9-19-64

Test Temp., °F	Mean Coefficient of Thermal Expansion, in./in./°F x 10^{-6}		
	Heating	Cooling	Average
77-392	7.66	7.66	7.66
77-572	7.84	7.84	7.84
77-752	8.08	8.08	8.08
77-932	8.44	8.44	8.44
77-1112	8.62	8.62	8.62
77-1292	8.92	8.92	8.92
77-1472	9.10	9.10	9.10
77-1562	9.25	9.25	9.25
77-1607	9.30	9.30	9.30

Test No. 4
Date: 9-30-64

Test Temp., °F	Mean Coefficient of Thermal Expansion, in./in./°F x 10^{-6}		
	Heating	Cooling	Average
77-392	7.75	7.58	7.67
77-572	7.90	7.75	7.85
77-752	8.20	8.12	8.16
77-932	8.47	8.41	8.44
77-1112	8.72	8.59	8.66
77-1292	8.94	8.81	8.88
77-1472	9.14	9.06	9.10
77-1562	9.28	9.18	9.23
77-1607	9.33	9.26	9.30

Test No. 5
Date: 11-2-64

Test Temp., °F	Mean Coefficient of Thermal Expansion, in./in./°F x 10^{-6}		
	Heating	Cooling	Average
77-392	7.66	7.66	7.66
77-572	7.79	7.79	7.79
77-752	8.04	8.04	8.04
77-932	8.35	8.35	8.35
77-1112	8.62	8.62	8.62
77-1292	8.86	8.86	8.86
77-1472	9.10	9.10	9.10
77-1562	9.21	9.21	9.21
77-1607	9.26	9.26	9.26

Test No. 6
Date: 11-7-64

Test Temp., °F	Mean Coefficient of Thermal Expansion, in./in./°F x 10^{-6}		
	Heating	Cooling	Average
77-392	7.50	7.50	7.50
77-572	7.69	7.69	7.69
77-752	8.00	8.00	8.00
77-932	8.38	8.38	8.38
77-1112	8.62	8.62	8.62
77-1292	8.83	8.83	8.83
77-1472	9.02	9.02	9.02
77-1562	9.18	9.18	9.18
77-1607	9.30	9.30	9.30

TABLE XVII: THERMAL EXPANSION DATA FOR Mo-TZM ALLOY[1]
(Nominal Composition: 0.5% Ti-0.08%Zr - Bal. Mo)

Specimen No.: MCN 1037-C-1
Date: 8-25-64

Test Temp., ^{o}F	Mean Coefficient of Thermal Expansion, in./in./$^{o}F \times 10^{-6}$		
	Heating	Cooling	Average
77-392	2.83	2.83	2.83
77-572	2.81	2.81	2.81
77-752	2.84	2.84	2.84
77-932	2.92	2.92	2.92
77-1112	2.94	2.94	2.94
77-1292	2.98	2.98	2.98
77-1472	3.02	3.02	3.02
77-1562	3.04	3.04	3.04
77-1607	3.02	3.02	3.02

Initial over-all length: 2.5600 inches.
Final over-all length: 2.5600 inches.

Specimen No.: MCN 1037-C-2
Date: 9-18-64

Test Temp., ^{o}F	Mean Coefficient of Thermal Expansion, in./in./$^{o}F \times 10^{-6}$		
	Heating	Cooling	Average
77-392	2.83	2.83	2.83
77-572	2.86	2.86	2.86
77-752	2.84	2.84	2.84
77-932	2.95	2.95	2.95
77-1112	2.94	2.94	2.94
77-1292	2.98	2.98	2.98
77-1472	3.02	3.02	3.02
77-1562	3.04	3.04	3.04
77-1607	3.04	3.04	3.04

Initial over-all length: 2.5595 inches.
Final over-all length: 2.5595 inches.

(1) Stress Relieved 1/4 Hour at 2200^{o}F

TABLE XVIII: THERMAL EXPANSION DATA FOR UNALLOYED ARC CAST TUNGSTEN[1]

Specimen No.: MCN 1038-C-1
Date: 9-28-64

Test Temp., ^{o}F	Mean Coefficient of Thermal Expansion, in./in./^{o}F x 10^{-6} Heating	Cooling	Average
77-392	2.25	2.25	2.25
77-572	2.23	2.23	2.23
77-752	2.30	2.30	2.30
77-932	2.33	2.33	2.33
77-1112	2.33	2.33	2.33
77-1292	2.35	2.35	2.35
77-1472	2.39	2.39	2.39
77-1562	2.41	2.41	2.41
77-1607	2.40	2.40	2.40

Initial over-all length: 2.5590 inches.
Final over-all length: 2.5590 inches.

Specimen No.: MCN 1038-C-2
Date: 9-21-64

Test Temp., ^{o}F	Mean Coefficient of Thermal Expansion, in./in./^{o}F x 10^{-6} Heating	Cooling	Average
77-392	2.25	2.25	2.25
77-572	2.28	2.28	2.28
77-752	2.33	2.33	2.33
77-932	2.33	2.33	2.33
77-1112	2.36	2.36	2.36
77-1292	2.38	2.38	2.38
77-1472	2.39	2.39	2.39
77-1562	2.41	2.41	2.41
77-1607	2.42	2.42	2.42

Initial over-all length: 2.5600 inches.
Final over-all length: 2.5600 inches.

(1) Stress Relieved 1 Hour at 2000^{o}F

TABLE XIX: THERMAL EXPANSION DATA FOR CARBOLOY GRADE 907
(Nominal Composition: 74% WC-20% TaC - 6% Co)

Specimen No.: MCN 1036-C-1
Date: 9-1-64

Test Temp., °F	Mean Coefficient of Thermal Expansion, in./in./°F x 10^{-6}		
	Heating	Cooling	Average
77-392	2.67	2.67	2.67
77-572	2.65	2.65	2.65
77-752	2.76	2.76	2.76
77-932	2.89	2.89	2.89
77-1112	2.94	2.94	2.94
77-1292	2.98	2.98	2.98
77-1472	3.05	3.05	3.05
77-1562	3.10	3.10	3.10
77-1607	3.10	3.10	3.10

Initial over-all length: 2.5600 inches.
Final over-all length 2.5600 inches.

Specimen No.: MCN 1036-C-2
Date: 9-24-64

Test Temp., °F	Mean Coefficient of Thermal Expansion, in./in./°F x 10^{-6}		
	Heating	Cooling	Average
77-392	2.67	2.67	2.67
77-572	2.71	2.71	2.71
77-752	2.76	2.76	2.76
77-932	2.89	2.89	2.89
77-1112	2.94	2.94	2.94
77-1292	2.98	2.98	2.98
77-1472	3.05	3.05	3.05
77-1562	3.12	3.12	3.12
77-1607	3.12	3.12	3.12

Initial over-all length: 2.5600 inches.
Final over-all length: 2.5600 inches.

TABLE XX: THERMAL EXPANSION DATA FOR CARBOLOY GRADE 999
(Nominal Composition: 97% WC - 3% Co)

Specimen No.: MCN 1035-C-1
Date: 9-2-64

Test Temp., °F	Mean Coefficient of Thermal Expansion, in./in./°F x 10^{-6} Heating	Cooling	Average
77-392	2.33	2.33	2.33
77-572	2.33	2.33	2.33
77-752	2.37	2.37	2.37
77-932	2.46	2.46	2.46
77-1112	2.54	2.54	2.54
77-1292	2.59	2.59	2.59
77-1472	2.64	2.64	2.64
77-1562	2.69	2.66	2.68
77-1607	2.68	2.64	2.66

Inital over-all length: 2.5597 inches.
Final over-all length: 2.5597 inches.

Specimen No.: MCN 1035-C-2
Date: 9-23-64

Test Temp., °F	Mean Coefficient of Thermal Expansion, in./in./°F x 10^{-6} Heating	Cooling	Average
77-392	2.33	2.33	2.33
77-572	2.33	2.33	2.33
77-752	2.41	2.37	2.39
77-932	2.52	2.46	2.49
77-1112	2.56	2.54	2.55
77-1292	2.66	2.59	2.63
77-1472	2,70	2.62	2.66
77-1562	2.73	2.66	2.70
77-1607	2.74	2.68	2.71

Initial over-all length: 2.5600 inches.
Final over-all length: 2.5600 inches.

TABLE XXI: THERMAL EXPANSION DATA FOR LUCALOX
(Nominal Composition: 99.8% Al_2O_3)

Specimen No.: MCN 1039-C-1
Date: 9-4-64

Test Temp., ^{o}F	Mean Coefficient of Thermal Expansion, in./in./$^{o}F \times 10^{-6}$		
	Heating	Cooling	Average
77-392	3.42	3.42	3.42
77-572	3.61	3.61	3.61
77-752	3.77	3.77	3.77
77-932	3.93	3.93	3.93
77-1112	4.06	4.06	4.06
77-1292	4.17	4.17	4.17
77-1472	4.28	4.28	4.28
77-1562	4.34	4.34	4.34
77-1607	4.33	4.33	4.33

Initial over-all length: 2.5593 inches.
Final over-all length: 2.5593 inches.

Specimen No.: MCN 1039-C-2
Date: 9-28-64

Test Temp., ^{o}F	Mean Coefficient of Thermal Expansion, in./in./$^{o}F \times 10^{-6}$		
	Heating	Cooling	Average
77-392	3.33	3.33	3.33
77-572	3.55	3.55	3.55
77-752	3.77	3.77	3.77
77-932	3.93	3.93	3.93
77-1112	4.06	4.06	4.06
77-1292	4.17	4.17	4.17
77-1472	4.28	4.28	4.28
77-1562	4.32	4.32	4.32
77-1607	4.33	4.33	4.33

Initial over-all length: 2.5582 inches.
Final over-all length: 2.5582 inches.

TABLE XXII: THERMAL EXPANSION DATA FOR ZIRCOA 1027
(Nominal Composition: Proprietary, 95.5% ZrO_2)

Specimen No.: MCN 1040-C-1
Date: 9-5-64

Test Temp., °F	Mean Coefficient of Thermal Expansion, in./in./°F x 10^{-6} Heating	Cooling	Average
77-392	4.92	4.50	4.71
77-572	4.99	4.56	4.78
77-752	5.06	4.67	4.87
77-932	5.16	4.79	4.97
77-1112	4.99	4.82	4.91
77-1292	5.05	5.01	5.03
77-1472	5.09	5.16	5.13
77-1562	5.10	5.24	5.17
77-1607	5.11	5.28	5.20

Initial over-all length: 2.5600 inches.
Final over-all length: 2.5595 inches.
Change in length: 0.0005 inch.

Specimen No.: MCN 1040-C-2
Date: 9-25-64

Test Temp., °F	Mean Coefficient of Thermal Expansion, in./in./°F x 10^{-6} Heating	Cooling	Average
77-392	4.92	4.50	4.71
77-572	4.88	4.40	4.64
77-752	5.02	4.59	4.81
77-932	5.13	4.70	4.92
77-1112	4.92	4.77	4.85
77-1292	4.92	4.90	4.91
77-1472	4.98	5.05	5.02
77-1562	4.99	5.17	5.08
77-1607	5.01	5.16	5.09

Initial over-all length: 2.5600 inches.
Final over-all length: 2.5595 inches.
Change in length: 0.0005 inch.

TABLE XXIII: THERMAL EXPANSION DATA FOR GRADE 7178
(Nominal Composition: Proprietary)

Specimen No.: MCN 1046-C-1
Date: 8-31-64

Test Temp., ^{o}F	Mean Coefficient of Thermal Expansion, in./in./^{o}F x 10^{-6}		
	Heating	Cooling	Average
77-392	2.33	2.33	2.33
77-572	2.39	2.39	2.39
77-752	2.49	2.49	2.49
77-932	2.55	2.55	2.55
77-1112	2.56	2.56	2.56
77-1292	2.64	2.64	2.64
77-1472	2.70	2.70	2.70
77-1562	2.69	2.69	2.69
77-1607	2.71	2.71	2.71

Initial over-all length: 2.5590 inches.
Final over-all length: 2.5590 inches.

- -

Specimen No.: MCN 1046-C-2
Date: 9-22-64

Test Temp., ^{o}F	Mean Coefficient of Thermal Expansion, in./in./^{o}F x 10^{-6}		
	Heating	Cooling	Average
77-392	2.33	2.33	2.33
77-572	2.39	2.39	2.39
77-752	2.45	2.45	2.45
77-932	2.52	2.52	2.52
77-1112	2.56	2.56	2.56
77-1292	2.64	2.64	2.64
77-1472	2.68	2.68	2.68
77-1562	2.69	2.69	2.69
77-1607	2.69	2.73	2.71

Initial over-all length: 2.5595 inches.
Final over-all length: 2.5595 inches.

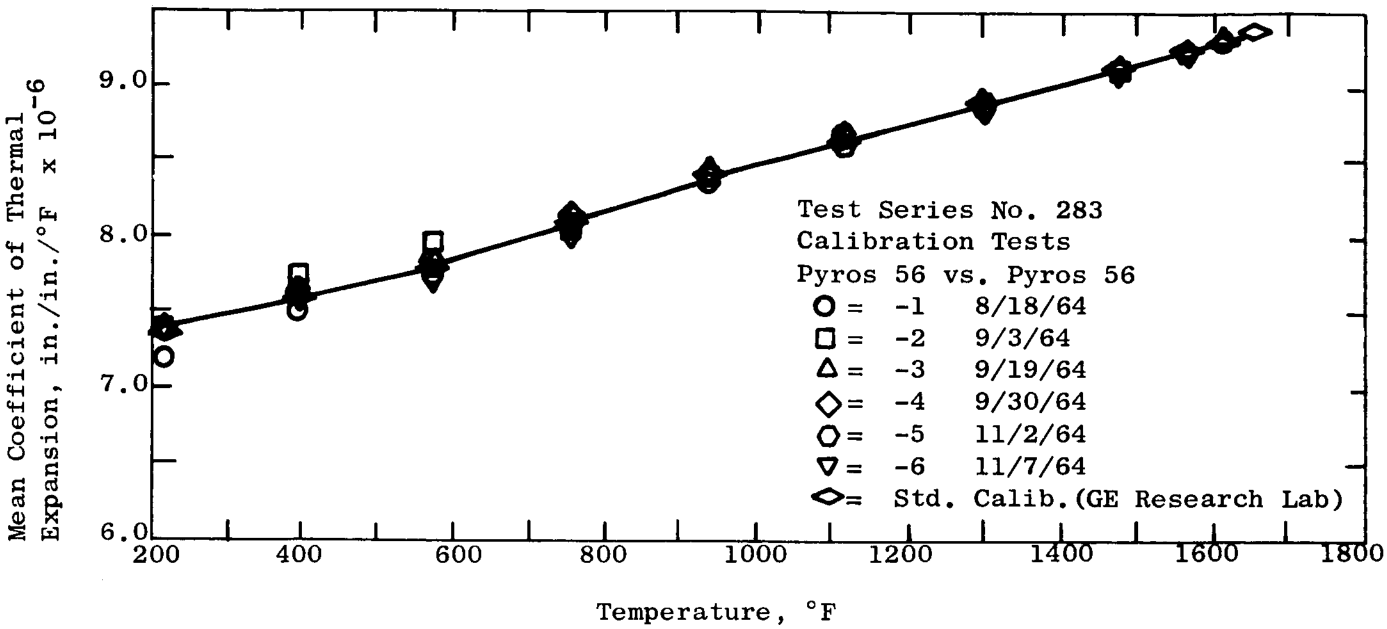


Figure 13. Mean Coefficient of Thermal Expansion of Pyros 56 Standard as a Function of Temperature.

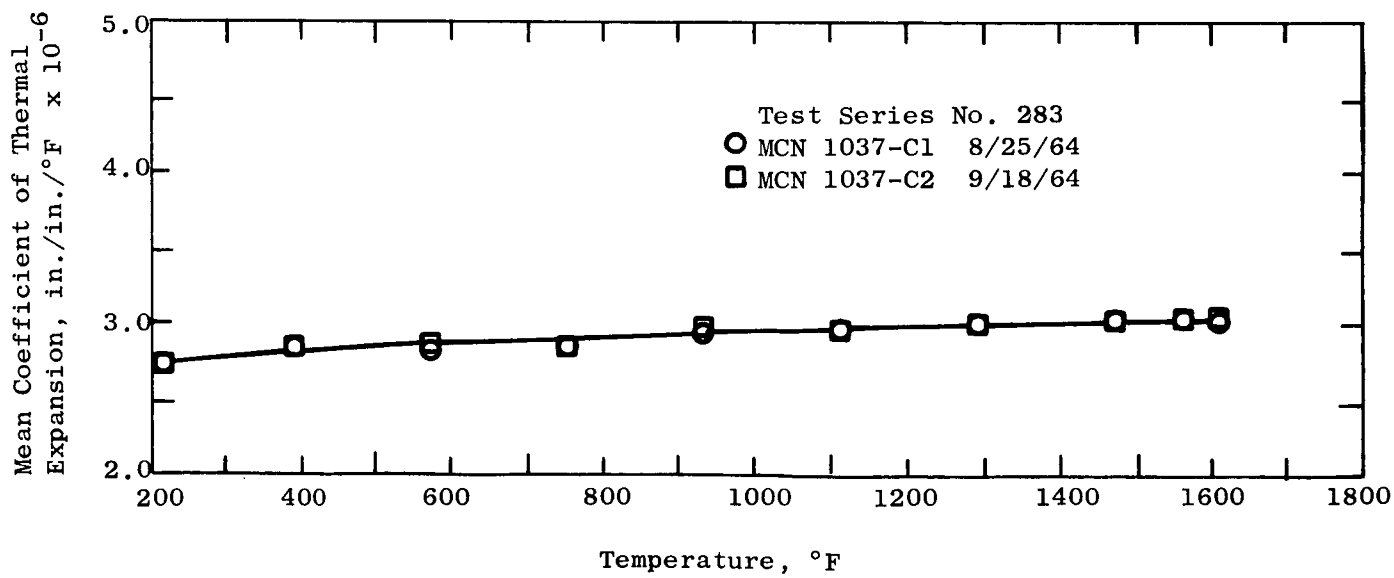


Figure 14. Mean Coefficient of Thermal Expansion of Mo-TZM Alloy (Stress Relieved 1/4 Hour at 2200°F) as a Function of Temperature.

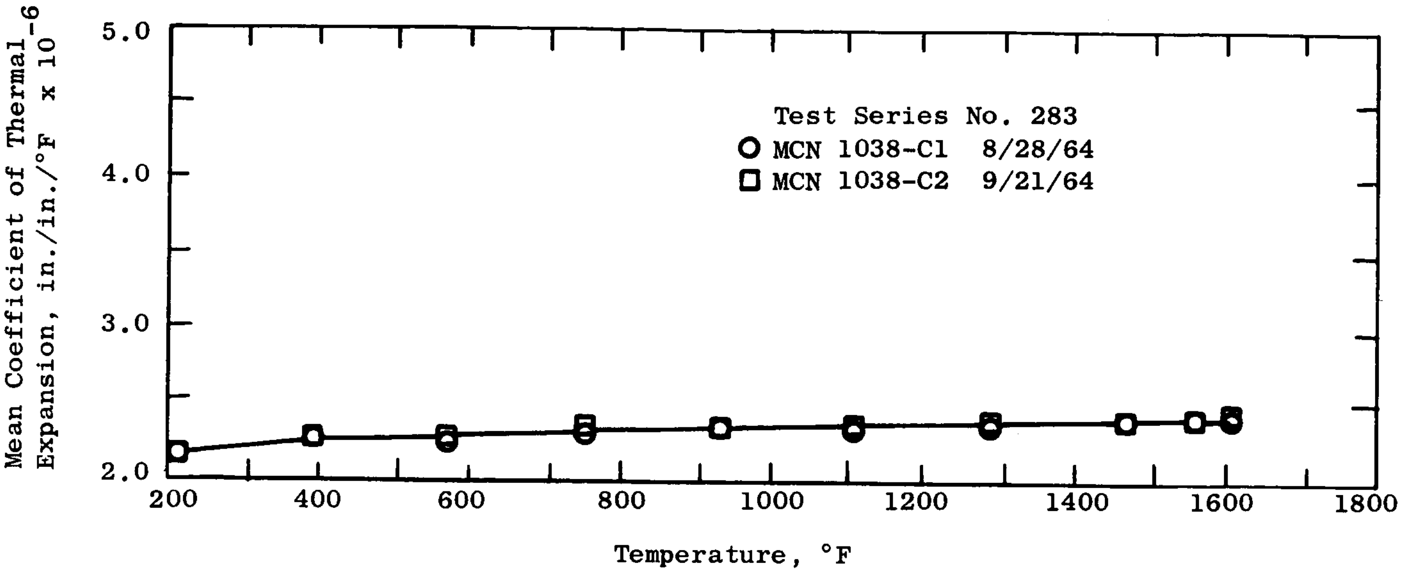


Figure 15. Mean Coefficient of Thermal Expansion of Arc Cast Tungsten (Stress Relieved 1 Hour at 2000°F) as a Function of Temperature.

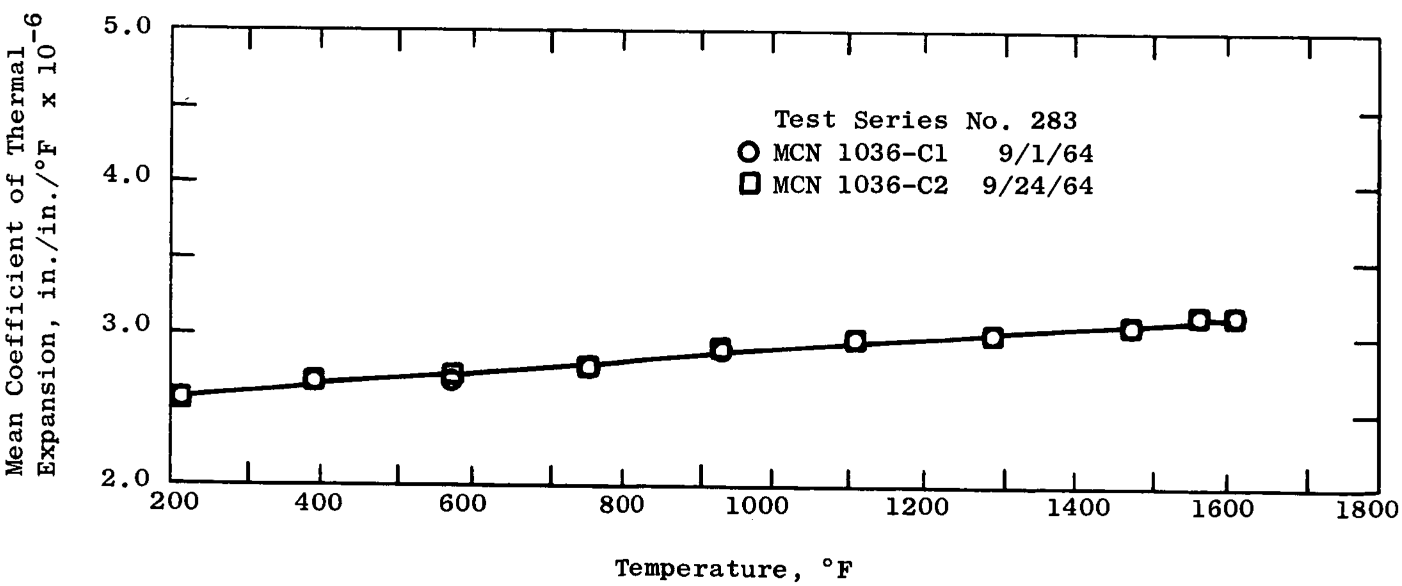


Figure 16. Mean Coefficient of Thermal Expansion of Carboloy Grade 907 as a Function of Temperature.

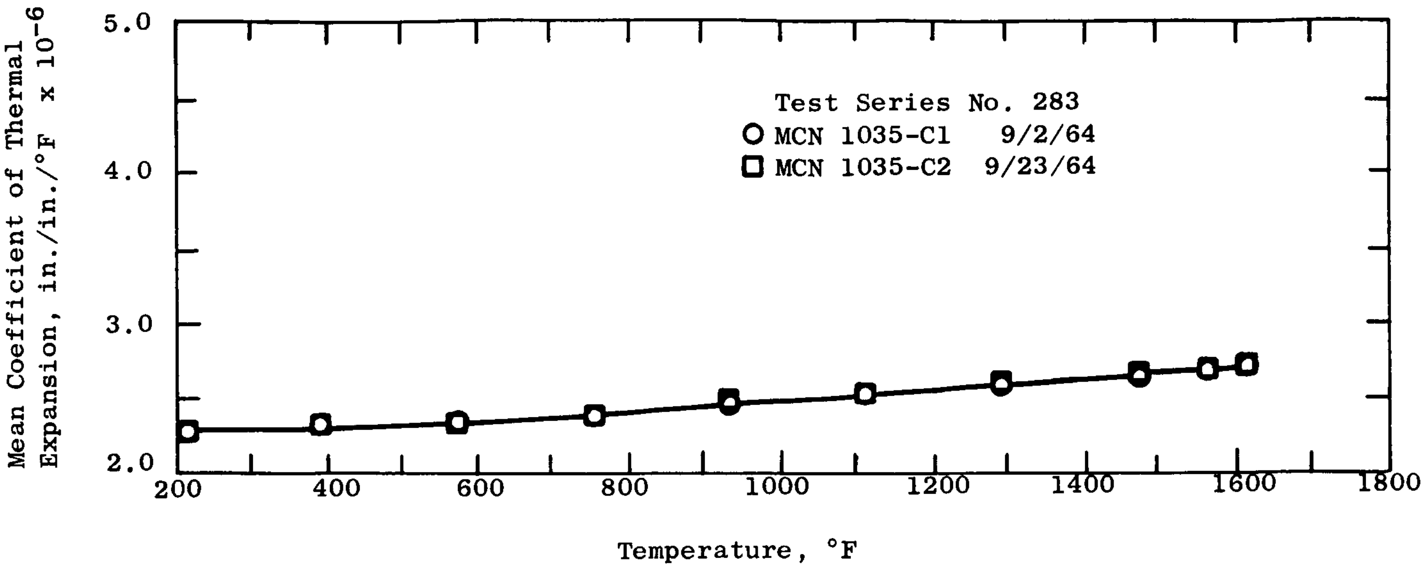


Figure 17. Mean Coefficient of Thermal Expansion of Carboloy Grade 999 as a Function of Temperature.

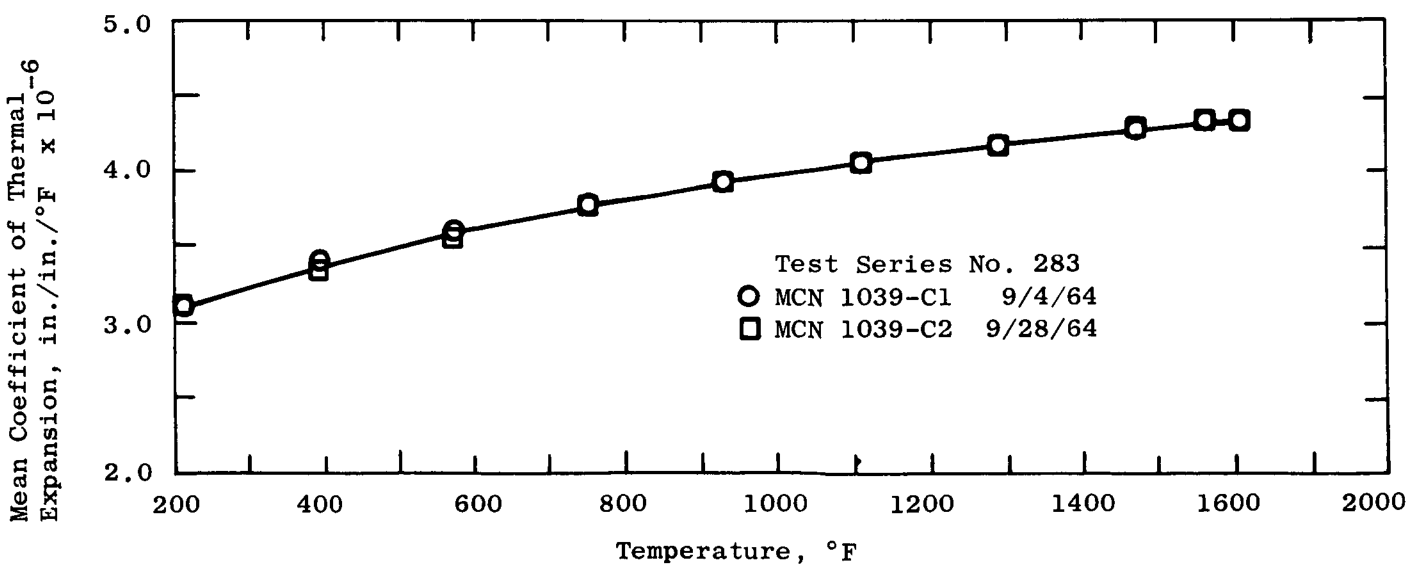


Figure 18. Mean Coefficient of Thermal Expansion of Lucalox as a Function of Temperature.

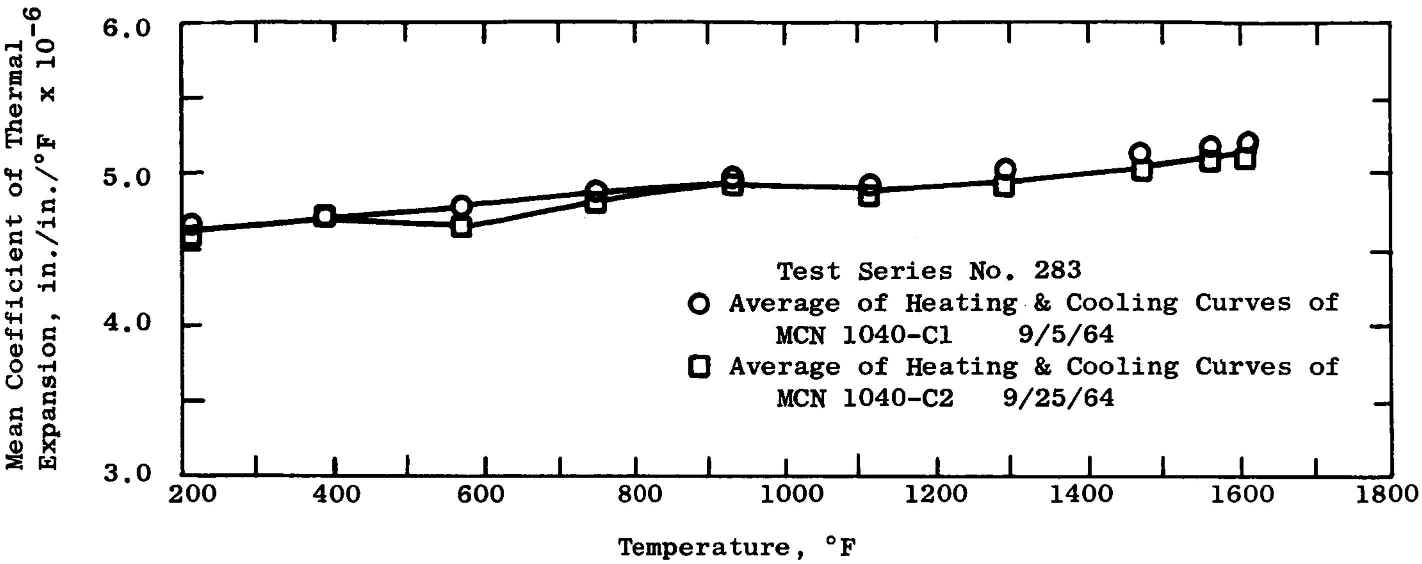


Figure 19. Mean Coefficient of Thermal Expansion of Zircoa 1027 as a Function of Temperature.

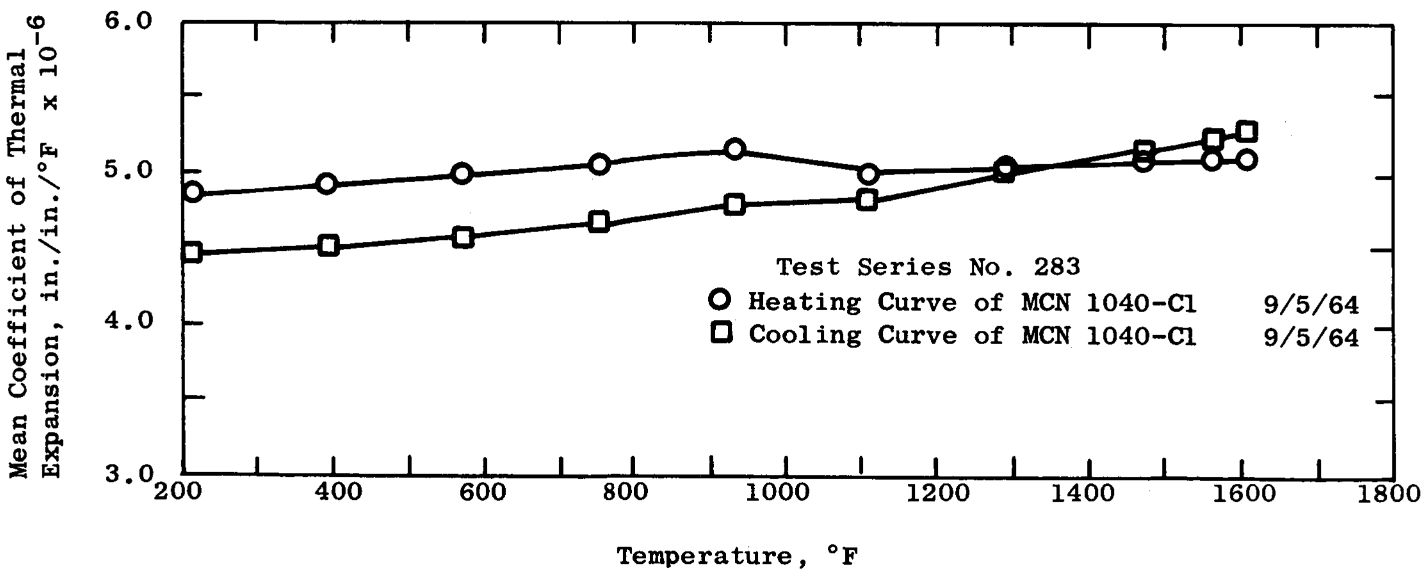


Figure 20. Mean Coefficient of Thermal Expansion of Zircoa 1027 as a Function of Temperature.

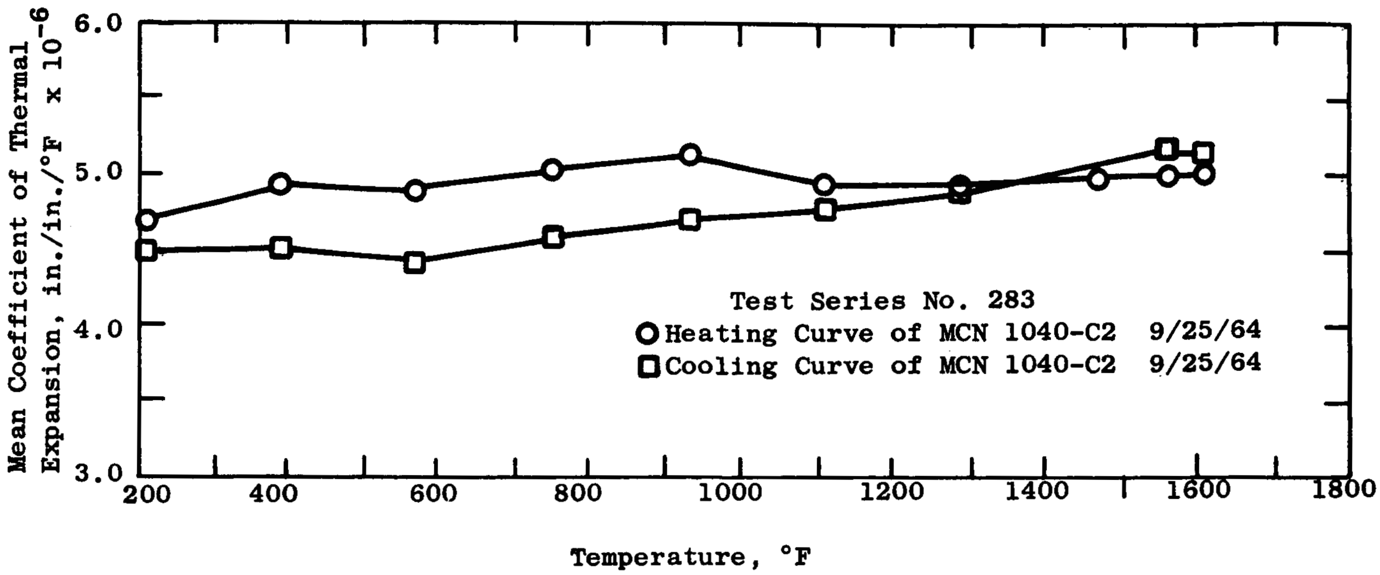


Figure 21. Mean Coefficient of Thermal Expansion of Zircoa 1027 as a Function of Temperature.

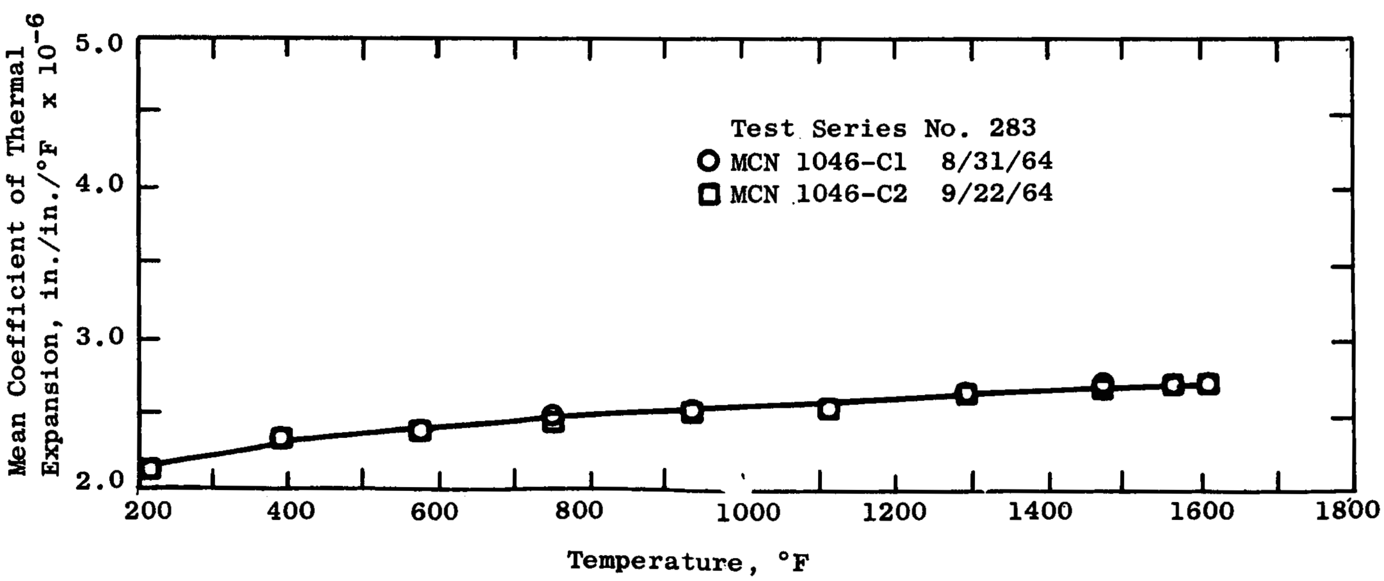


Figure 22. Mean Coefficient of Thermal Expansion of Grade 7178 as a Function of Temperature.

VI. FUTURE PLANS

The summary which follows enumerates the steps to be pursued during the succeeding quarter to implement this study.

1) Procurement of the compression test specimens will be completed.

2) Evaluation of the corrosion test specimens which were exposed to potassium for 1,000 hours at 1600°F, 1200°F, and 800°F under isothermal conditions will be initiated.

3) The second 1,000-hour dimensional stability test, incorporating thirteen materials at 800°F, will be completed and a third 1,000-hour test at 1200°F and 1600°F will be initiated.

4) The thermal expansion test program will be completed and the hot hardness and compression test programs will be initiated.

5) The high vacuum friction and wear test rig components will be received, assembled, and checked out.

6) The liquid potassium friction and wear test rig components will be received and assembly will be initiated.

7) The potassium distillate in the hot trap will be analyzed, the distillate will be hot trapped for 50 hours at 1300°F to 1400°F, and the hot trapped distillate will be re-analyzed in preparation for the first checkout test of the friction and wear test rig facility.

8) Drawings of the wetting test facility will be submitted to NASA and procurement of component parts will be initiated.

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